EFFECT OF FLOTATION REAGENTS ON THE ADSORPTION OF GOLD BY ACTIVATED CARBON

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DECLARATION

I declare that this report is my own work. It is being submitted to the degree of Master of Science in Engineering to the University of the Witwatersrand, Johannesburg. It has not been submitted before.

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(Signature of Candidate)

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ABSTRACT

The use of activated carbon in gold extraction is one of the most established methods for the recovery of gold from a leach solution. This is due to its lower operational cost and its ability to produce gold bullion with good purity compared to other methods such as cementation. Flotation of sulphide gold ores is commonly used as one of the beneficiation processes before leaching. However, the type and dosage of flotation reagents can affect downstream processes due to their fouling effect on granular activated carbon. This study aims to investigate the fouling effect of the most widely used flotation reagents namely potassium amyl xanthate and methyl isobutyl carbinol on the loading capacity and adsorption kinetics of gold onto the activated carbon. Experiments to determine fouling effect of activated carbon caused by flotation reagents were conducted in five steps. These included preparation of a gold standard solution, fouling of the activated carbon, carbon activity test, solution sample analysis and carbon analysis.

The results indicated that fouling of the activated carbon led to a lower adsorption rate and loading capacity of gold. The fouling due to potassium amyl xanthate was found to be more significant compared to the fouling due to methyl isobutyl carbinol. In both systems, the effect was more pronounced with a high concentration of flotation reagents and a lower concentration of the activated carbon in the system. This suggests that the use of a high amount of flotation reagents should subsequently be complimented by high carbon addition in order to minimize the effect of the flotation reagents during the gold adsorption stage. This however, will effectively have an impact on the operational costs.

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CHAPTER ONE

INTRODUCTION

1.1 General Introduction

Gold processing of free milling ores by conventional cyanidation can achieve an extraction of 90% and above compared to 50% to 80% for refractory ores (Celep *et al*., 2009). The refractory nature of gold is often associated with gold being enclosed within insoluble minerals, presence of carbonaceous materials (preg robbing ores) and gold being enclosed within soluble minerals to cyanide (chalcopyrite, pyrite) (Van den Berg, 2000; Srithammavut, 2008).

In order to achieve high extraction of gold from carbonaceous materials or preg robbing ores, pre-treatment of the ore is required. Treatment may be done by gravity concentration followed by intensive cyanidation of concentrates, application of carbon in the leach process with highly concentrated activated carbon, and roasting of the ore to burn all carbonaceous materials. For the refractory nature of gold ores often associated with gold finely disseminated in sulphide minerals such as pyrite, conventional milling can liberate the pyrite from the gangue allowing a low mass concentrate to be produced by a process such as flotation. The route that is mostly applied on this type of ore includes; comminution, classification, flotation, pre-treatment of flotation concentrate and leaching. Comminution aims to reduce the size of solid particles to increase surface area for chemical reaction, whereas classification aims to prevent coarser particles from going to the flotation process.

Flotation is commonly used as an intermediate step to allow one of the pre-treatment processes such as pressure oxidation, roasting, fine/ultra-grinding to be carried out on a smaller and concentrated fraction of materials (Marsden and House, 2006). Pre-treatment of flotation concentrate is necessary due to poor results in leaching of the concentrate as the cyanide lixiviant is unable to contact the gold locked or included within the pyrite. The pre-treated concentrates and in some cases along with the flotation tailings, are transferred to the carbon in pulp (CIP) or carbon in leach (CIL) processes where gold is dissolved and adsorbed onto the activated carbon (Marsden and House, 2006). The traditional pre-treatment approach for such refractory concentrate has been to liberate the locked gold by chemically destroying the pyrite through

oxidation. Whilst such oxidative reactions are metallurgically sound and are capable of achieving high metal recoveries the environmental aspects of treating the reaction products can alter the economics of the process. An alternative, applicable to the liberation of disseminated gold from the host mineral, is to continue the grinding process to further reduce the particle size of the host mineral thereby exposing part of the gold surface for contact with cyanide solution. A benefit of this ultra fine grinding technique is that the host mineral is not destroyed in an oxidative chemical reaction. Ultra fine grinding usually fine grinds to a range of l μm to 10 μm, and impart a significantly increased surface area which increases chemical reactivity.

Although the leaching of ultra fine flotation concentrate has shown a great improvement in gold extraction, the type and dosage of flotation reagents can affect downstream processes due to their fouling effect on granular activated carbon. In the flotation circuit, several reagents such as collectors, frothers, modifiers, activators and depressants are used to maximize the recovery of all the gold bearing sulphide minerals (Marsden and House, 2006; Dunne, 2005). The presence of residual flotation reagents has proved to be a major problem during leaching of gold and adsorption of gold by activated carbon. In the CIP process, the conventional processing of gold bearing ores requires a cyanidation step and subsequent adsorption of the resulting gold on activated carbon. Although activated carbon is ideal for the adsorption of gold cyanide complex, it is unselective and will adsorb, with various degrees of efficiency, most of the dissolved inorganic or organic compounds present in the leach solution. Thus, the quantity of gold that can be adsorbed is diminished. In general, the gold sorption efficiency onto the activated carbon is affected by a number of physical and chemical factors including carbon type and size, mixing efficiency, gold and cyanide concentration, presence of other metal ions and complexes in leach solution, solution pH and temperature, ionic strength and carbon fouling (Marsden and House, 2006). The carbon fouling is defined as the adsorption or accumulation of unwanted organic and inorganic species on the surface or within the pores of the activated carbon (Marsden and House, 2006; Dunn and Fisher, 2001). Inorganic fouling agents consist of calcium carbonate, silica and base metal complexes present in the leach solution. Many inorganic fouling agents can be removed from the activated carbon by a downstream process of acid washing (Dunn and Fisher, 2001). Of greater concern are the organic fouling agents, which consist of oils, greases, and process chemicals. These organic fouling agents are present in most carbon adsorption circuits as a result of their addition or spillage during mining, milling, flotation, and equipment maintenance

operations. It has been reported that the most serious carbon deactivation is caused by flotation reagents (La Brooy *et al.* 1986; Salarirad *et al*., 2010)

The flotation reagents such as collectors, frothing agents, flocculants, and oils are some of the main organic foulants present in the leach solution and subsequently affect the kinetic activity and loading capacity of the carbon. This is due to film formation on the external surface of activated carbon which acts as a barrier by reducing mass transfer of gold into the pores of activated carbon (Salarirad *et al*., 2011). Flotation reagents such as xanthate and methyl isobutyl carbinol, do not penetrate into the pores of activated carbon and only form a layer on the superficial surface of the carbon granules that diminish mass transfer, resulting in reduced sorption kinetics and hence affect the adsorption rate and not the loading capacity of gold onto activated carbon (Salarirad *et al*., 2010). Other studies (Fisher, 2000; Ross, 1993; Mahapatra, 2009) have shown that the flotation reagents have the potential to affect both the adsorption rate and loading capacity of gold onto activated carbon. This is due to blocking of the pores of activated carbon and hence affecting the mass transfer of gold onto activated carbon (Mahapatra, 2009). However, studies by Petersen *et al*. (1997) have shown that, there is no blocking of activated carbon pores by large molecules of flotation reagents due to their difficulty in diffusing into the mesopores and micropores of activated carbon. The study concluded that, there is only a competition for active sites on the surface of activated carbon between organic compounds and gold. This competition affects the overall activity of activated carbon, and the effect is more critical when there is high loading of organic compounds on the surface of activated carbon.

1.2 Problem Identification

The adsorption rate of gold cyanide onto activated carbon (commonly referred to as activity) controls the solution tails in a carbon in pulp (CIP) circuit. The activity determines the amount of carbon required, contact time (tank size), gold inventory (lockup), and overall efficiency of the plant (Mahapatra, 2009; La Brooy *et al*., 1984). Maintaining high carbon activity is vital to the efficiency of any Carbon-In-Leach or Carbon-In-Pulp process. Reduced activity can lead to longer residence times or gold losses in the tails. As such, it is important to investigate the effect of the most widely used flotation reagents on the adsorption of gold onto activated carbon. Furthermore, the combinational effect of reagents, including collector (potassium amyl xanthate)

and frother (methyl isobutyl carbinol), on the kinetics of gold adsorption and loading capacity onto granulated activated carbon needs to also be evaluated. The results from this project will contribute to the advancement in knowledge required to determine optimum concentration of flotation reagents which will have minimum fouling effect on activated carbon. This will subsequently lead to high production of gold by preventing the loss of gold (solution) to the tailing dams due to problems caused by the fouling of activated carbon arising from the flotation reagents.

1.3 Aims and Objectives of the study

The main aim of the research work is to investigate the fouling effect of the most widely used flotation reagents used in the gold industry namely potassium amyl xanthate (PAX) and methyl isobutyl carbinol (MIBC) at variable concentration and under typical gold plant conditions, on the loading capacity and adsorption kinetics of gold onto the activated carbon.

In order to address the aim, the research seeks to answer the following question:

- What effect do the different levels of concentration of flotation reagents have on the maximum amount of gold that can be adsorbed onto activated carbon?
- What effect do the different levels of concentration of flotation reagents have on the rate of gold adsorption onto the activated carbon?

1.4 Research Methodology

The research methodology involved the following tasks: Literature review, experimental design and laboratory testwork, results and discussion, conclusions and recommendations.

CHAPTER TWO

LITERATURE REVIEW

2.1 Gold and its Occurrence

Gold with an atomic number of 79, atomic weight of 197 and with a chemical symbol of Au, is one of the most valuable minerals that is found on the Earth's crust (Marsden and House, 2006). The average concentration of gold in the Earth's crust is 0.005ppm which is very low compared to other minerals e.g. silver and copper (Marsden and House, 1992; Srithammavut, 2008). Gold has physical and chemical properties such as specific gravity of 19.3, melting point of 1064^0C and boiling point of 2856^0C (Marsden and House, 1992). Gold is used for jewellery making, as a medium of exchange and in electronic industry where corrosion resistance is a problem (Kasongo, 2008).

It occurs either as free gold (liberated gold) or gold that is locked within other minerals and the separation is based on their reaction to cyanide leaching (Celep *et al*., 2009). Free milling ores (liberated gold) do not require treatment before leaching while refractory ores require treatment before leaching (Gray, 1999). Gold processing of free milling ores by conventional cyanidation can achieve an extraction of 90% and above compared to 50% to 80% for refractory ores (Celep *et al*., 2009).

In the past gold has been recovered by amalgamation which involves the use of mercury (Marsden and House, 1992). The method has failed due to poor recoveries and the associated risk to the environment and health. The current methods for recovery of gold include the use of a gravity concentrator, flotation and leaching by chemical cyanide (Srithammavut, 2008).

2.2 Extraction of Gold from Ores

The extraction of gold depends mostly on the nature of the ore to be processed (Marsden and House, 2006; Fisher, 2000). The route that is mostly applied for free milling ores includes comminution, classification and leaching. This is different from refractory ores due to nature of

the ore (complex ores). The route that is mostly applied for refractory ores includes; comminution, classification, flotation, pre-treatment of flotation concentrate and leaching (Fisher, 2000). Figure 2.1 shows the extraction route used to process refractory ores.

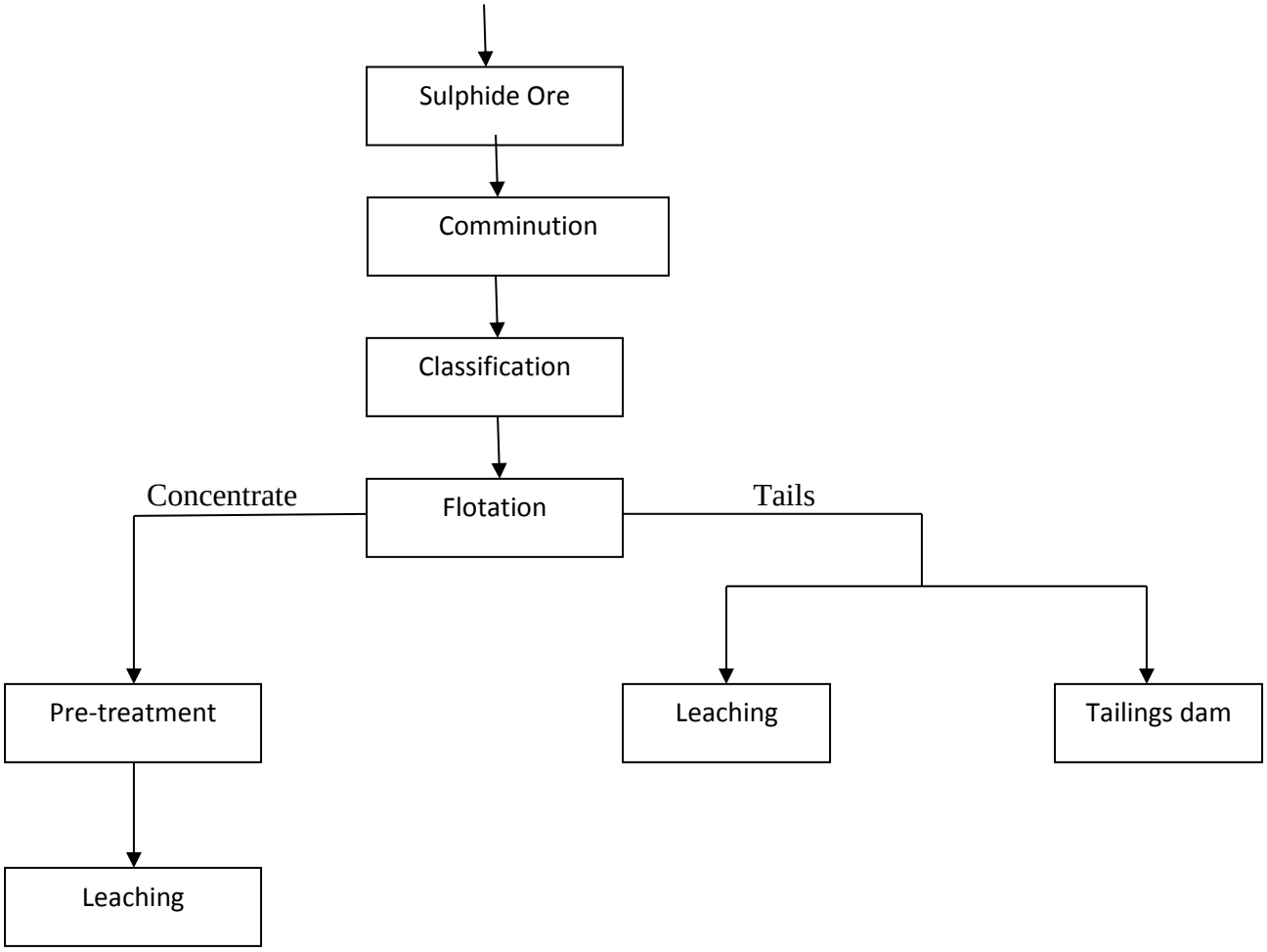


Figure 2.1: Flow sheet of gold extraction from sulphide ore (Fisher, 2000)

2.3 Comminution

Comminution aims to reduce the size of solid particles so as to increase the surface area for chemical reaction in subsequent processes (Wills, 2006). The process involves the uses of crushers and grinding mills (Gupta, 2003). The crushing process aims to reduce the size of run of mine (ROM) to such a level that grinding can be carried out, whereas grinding aims to produce fine particles of valuable mineral which can be separated in a certain process such as leaching (Wills, 2006).

2.4 Classification

Classification is a method aimed at separating a mixture of materials into different products (Wills, 2006). The separation is based on the velocity with which the grains fall through a fluid medium. This prevents the treatment of different sizes of particles within the same circuit. A study by Wills (2006) shows the effect of treating finer and coarser particles within the same system. It results in poor recoveries and leads to high loss of valuables in the tailings dam. Therefore, classification prevents coarser particles from going to the flotation process. The study by O' Connor *et al*. (1994) concluded that, in order to achieve high recovery in a flotation circuit; a classifier should be efficient enough to send 80% passing of 75 μm to the flotation circuit.

2.5 Flotation

Flotation is the process which utilises differences in surface properties of wanted and non-wanted minerals (Wills, 2006). The separation of the required minerals from the ore is achieved by the difference in surface properties (Gupta, 2003).

According to Wills (2006), the process of recovering gold from the ore by flotation, consists of mechanisms such as:

- Attachment of valuable minerals to air bubbles
- Chemistry of carrying valuable particles in the pulp
- Physical attraction between particles in the froth that stick to air bubbles

Valuable mineral attachment to air bubbles is considered as the main mechanism during flotation (Wills, 2006). It follows after the separation of valuable minerals from gangue minerals. The separation of minerals depends on the degree of physical entrapment and entrainment (Gupta, 2003). Entrainments of gangue minerals affect the quality of valuable concentrate. In order to increase the recovery of the valuable concentrate, more than one flotation stage is required. The study by Wills (2006) showed that an increase in number of flotation stages results in an increase in the recovery of valuable material and hence less tails.

Flotation is divided into two types, named direct and reverse flotation (ed. Darling, 2011). In gold flotation, direct flotation involves the removal of gold as froth while gangue minerals remain in the pulp. Reverse flotation involves the removal of gangue minerals as froth while gold minerals remain in the pulp. Gold particles to be selected or attached to the air bubbles need to have hydrophobic properties. Hydrophobic properties refer to the capability of minerals to be attached to air bubbles and become water repellent (Wills, 2006). In order for the air bubbles to carry gold minerals, it is necessary to form a stable froth (ed. Darling, 2011). This will prevent the bursting and dropping of gold particles once the bubbles get to the surface. The addition of flotation reagents such as frothers have been shown to prevent the bursting of bubbles and subsequent dropping of valuable minerals (Wills, 2006). In addition, there are forces which act on the valuable surfaces. These forces tend to separate a particle and a bubble interface. Figure 2.2 demonstrates the contact angle between bubble and gold particle in an aqueous medium (Wills, 2006).

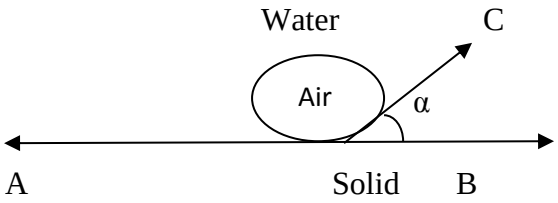


Figure 2.2: The contact angle between bubble and gold particle in an aqueous medium (Wills, 2006)

The study by Wills (2006) showed that the work required to break the contact between gold particles and air is given by the equation:

$$W = C\,(1 - \cos\alpha) \quad \ldots\ldots\ldots\

2.5.1 Flotation Reagents

In order to achieve the recovery of gold minerals by flotation, a mixture of chemical compounds or flotation reagents are added to the circuit. Reagents may be grouped based on their function during the process. They include:

- Collectors
- Frothers
- Modifiers

Collectors

Collectors are organic compounds added to impart the degree of hydrophobicity of a gold mineral (ed. Darling, 2011). They undergo chemical reaction with the surface of a gold mineral. Collectors mostly consist of two parts named polar and non-polar (hydrocarbon) (Gupta, 2003). The surface of gold mineral is attacked by the polar part of a collector to make it hydrophobic.

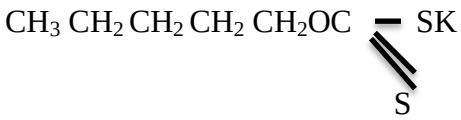
Collectors may either be ionising or non-ionising compounds (Wills, 2006). Ionising compounds dissolve into ions in the presence of water while non-ionising do not dissolve in water (Gupta, 2003). They only cover the surface of valuables with a thin layer. This layer makes minerals to become hydrophobic and hence float to the surface (Wills, 2006).

The main collector used in the gold industry is potassium amyl xanthate (PAX) (O' Connor *et al*., 1994; Wills, 2006; Gupta, 2003). The addition of PAX is done in small quantities to avoid the flotation of unwanted minerals since the unwanted minerals tend to affect the quality of the final concentrate (ed. Darling, 2011). PAX is a type of sulphydryl collectors used to treat gold ore associated with sulphide minerals. This is due to their high selectivity as a result of reaction with the sulphide surface (Avotins *et al*., 1994). Moreover, PAX has a longer hydrocarbon chains which increase the degree of hydrophobicity of a mineral (Wills, 2006).

Potassium amyl xanthate is a water soluble chemical with a molecular weight of 164.289 g/mol. It is formed from a reaction of alcohol, carbon disulphide and potassium hydroxide (Wills, 2006). The reaction can be written as:

$$ROH + KOH + CS_2 = ROCSSK + H_20$$

Where R stands for hydrocarbon group and comprise of 1 to 6 atoms of carbon, C, K, OH, O, S, and H_2O stands for carbon, potassium, hydroxyl group, oxygen, sulphur and water. The study by Wills (2006) shows the molecular formula of PAX and that it consists of elements such as carbon, hydrogen, oxygen, sulphur, sodium and potassium. The chemical structural formula is given as:

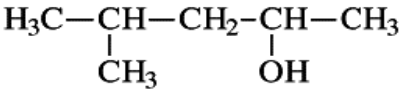


Although PAX is mostly used in gold plants, it has storage problem. It tends to lose its selectivity as a result of oxidation with time which then subsequently influences the recovery of valuable metals (Wills, 2006).

Frothers

These are reagents which are added to strengthen and stabilize the loaded bubble (Gupta, 2003). In order to prevent the bursting of the bubble, a good frother should be used to facilitate the transfer of gold particles from the cell to the cell launder (Wills, 2006). Methyl isobuyl carbinol (MIBC) is a type of frother used in gold flotation plants.

MIBC is an organic chemical compound with a molecular weight of 102.2 g/mol (Wills, 2006). It has a density of 0.808 g/mol, and boiling point and freezing point of – 90 ^{0}C and 132 ^{0}C respectively. It belongs to a group of aliphatic alcohol which consists of 6 to 8 atoms of carbon. The study by Wills (2006) shows the chemical composition of MIBC and it consists of carbon, hydrogen, and hydroxyl group. The chemical structural formula is given as:



MIBC is mostly used in flotation of gold ores due to its high kinetic activity, selectivity and ability to transfer loaded bubble from the cell to the cell launder (Wills, 2006; Gupta, 2003). The study by O' Connor *et al*. (1994) concluded that MIBC, is mostly used when selectivity is required and when dealing with copper-gold ores.

Both PAX and MIBC consist of polar and non-polar part. The difference between them is that the polar part of PAX has an attraction to the mineral of interest (gold mineral), whereas the polar part of MIBC has an attraction for water molecules only (Gupta, 2003). This indicates that the non-polar part of PAX will remain in contact with pulp, whereas the polar part of MIBC will remain in contact with pulp. Figure 2.3 shows the action of frother and a collector.

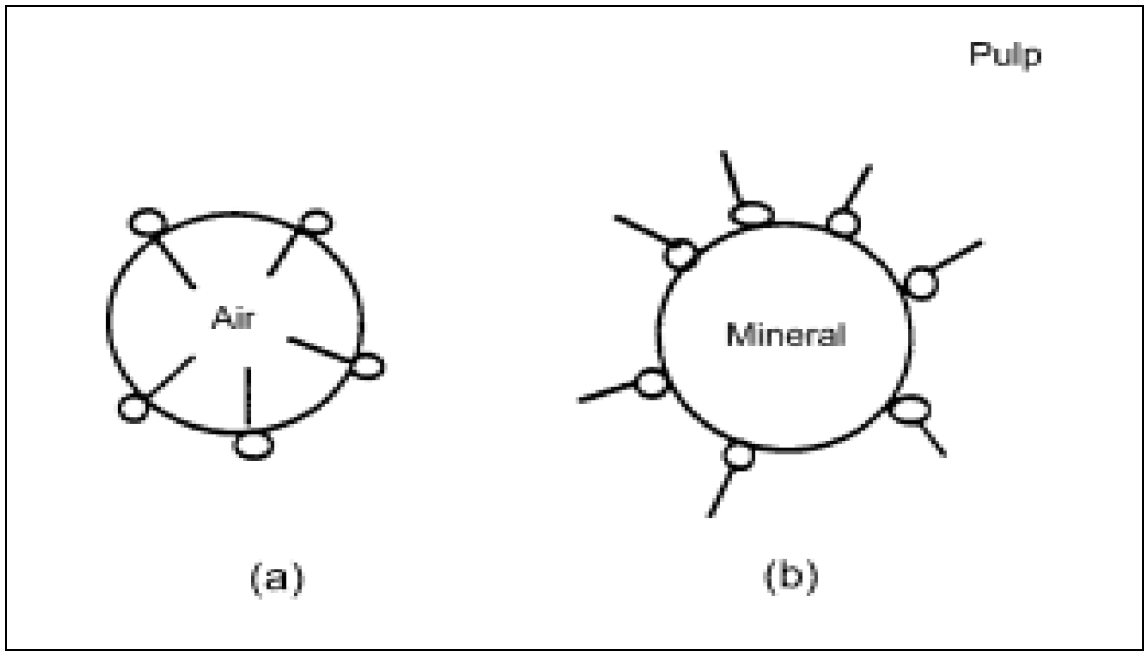


Figure 2.3: (a) Action of frother (MIBC) on air bubble (b) Action of Collector (PAX) on gold mineral surface (Gupta, 2003)

Whereby Polar part of a reagent

Non-polar part of a reagent

Modifiers

These are reagents which are added to improve the selectivity of gold minerals or to manipulate the means by which collectors attach to the surface of a gold particle (Gupta, 2003). These consist of pH modifiers, depressants and activators.

a) pH modifiers

pH modifiers are added to modify the action of collectors so as to prevent the flotation of unwanted minerals (Gupta, 2003). Minerals tend to form a negative charge in alkaline media and a positive charge in acidic media (ed. Darling, 2011). Therefore, the change of charge due to pH modification influences the attraction of collectors to a mineral surface. The main pH modifiers used in gold industry are caustic soda, soda ash and lime (Wills, 2006).

The use of lime as a pH modifier however, tends to have a negative effect during gold flotation (Wills, 2006). Calcium ions cause the formation of a film on the surface of the gold particles and this influences the effect of collectors (Wills, 2006). The best alternative is the use of soda ash and sodium hydroxide. This is due to less or negligible effect on the surface of gold particles (ed. Darling, 2011).

b) Depressants

These are chemicals used to increase the quality of the concentrate (Wills, 2006). They facilitate the selectivity of gold minerals and hinder (depress) the flotation of unwanted minerals. The depression arises as a result of changing the properties of unwanted minerals to become more hydrophilic (Bradshaw *et al*., 2005). The major types of depressants used in gold industry are lime, cyanide and carboxymethyl cellulose. Lime is added to depress pyrite during the flotation of gold ores associated with chalcopyrite and it operates at a pH of 10.5 (Wills, 2006). However, the addition of lime should be well monitored as it tends to reduce the xanthate efficiency (ed. Darling, 2011).

c) Activators

These are chemicals that promote the adsorption of collectors onto the mineral surface (Wills, 2006) and are applied to minerals which cannot float in aqueous solution or have been depressed by the action of depressants (ed. Darling, 2011). These chemicals are soluble salts which ionize in aqueous solution and lead to the formation of ions which subsequently react with the mineral surface. Adams (2005) showed that the addition of copper sulphate (activator) led to an increase

in the recovery of gold by 5 %. This was attributed to the deposition of copper sulphide on the mineral surface which subsequently led to the formation of insoluble copper xanthate making the gold mineral hydrophobic.

2.6 Gold Cyanidation Process

Cyanidation has become one of the established methods for the extraction of gold. This is due to higher recoveries compared to mercury amalgamation which has a recovery of less than 60% (Srithammavut, 2008; Marsden and House, 2006).

Cyanidation involves the dissolution of valuable minerals present in the ore in sodium cyanide containing solution at a pH of 10-11 (Davidson, 1992; Marsden and House, 2006). At pH below 9, sodium cyanide reacts with hydrogen and produces a poisonous gas of hydrogen cyanide whereas a pH above 11 may result in inorganic fouling of activated carbon. The equation for the leaching of gold is known as the Elsner's Equation (Srithammavut, 2008; Marsden and House, 2006). It is written as follows:

$$4Au + 8CN^- + O_2^- + 2H_2O \longrightarrow 4Au(CN)_2^- + 4OH^-$$

2.7 Gold Adsorption by Activated Carbon

Activated carbon is a good adsorbent which is why it is used in many applications such as in ground water remediation, treatment of drinking water, air purification, and removal of heavy metal pollutants from waste water (Bansal *et al*., 1998). However, activated carbon is mostly used in gold plants for the recovery of gold from leach solutions (Marsden and House, 2006).

Although the leached gold from the cyanidation process can be recovered by various methods such as ion exchange resins, cementation, and electrowinning (Kasongo, 2008), activated carbon has however, become one of the established methods for gold recovery. This is due to the production of high purity product (a purer solution that is electrowon to yield a purer bullion), and lower operating cost compared to other methods (Marsden and House, 2006). The use of activated carbon does not necessarily require the use of a thickener and filtration for the solid liquid separation step or the addition of zinc such as in the cementation method. This has a huge

impact on the operational costs since the presence of a thickener and filtration circuit increases the operating costs (Kasongo, 2008).

Activated carbon has high adsorption efficiency as a result of the highly developed pore structure, large surface area and the presence of surface functional groups. These characteristics are obtained from partial combustion and thermal decomposition of carbonaceous materials. It is noted that, 1 gram of activated carbon has a surface area of more than 800 m^2 as determined by gas adsorption (Fisher, 2000; Mahapatra, 2009; ed. Adams, 2005). Activated carbon is characterized based on the size (Jankowska *et al*., 1991). Powdered activated carbon has higher activity during the adsorption of valuable metals compared to granular activated carbon. This is due to the presence of larger pore diameters (Jankowska *et al*., 1991). Although granular activated carbon is weaker compared to powdered activated carbon, it is however mostly used in gold plants due to its regeneration capability (Mahapatra, 2009). A study by Davidson (1992) showed that powdered activated carbon cannot be regenerated and this increases the operating cost.

2.8.1 Manufacture of Activated Carbon

The manufacture of activated carbon consists of two stages namely pyrolysis or carbonisation and activation or gasification. The quality of the final product depends on the characteristics of raw material, applied activation temperature and additives used (Mahapatra, 2009). The most common raw materials used include peat, wood chips, vegetable products and coconut shells (Mahapatra, 2009; Van den Berg, 2000; Coetzee, 1996).

a) Carbonisation

This process involves the production of char from carbonaceous materials (Coetzee, 1996). The heating of carbonaceous materials takes place in an inert atmosphere at a temperature of about 300 - 600 °C (McDougall and Fleming, 1987). The process aims to remove the impurities such as volatile matter and gases which include carbon dioxide, carbon monoxide, and methane. The process leads to the formation of a porous char which has a poor adsorption ability as a result of the small surface area (Mahapatra, 2009).

b) Activation

The process involves the development of a porous char structure (Fisher, 2000; Rees, 2000). The carbonisation product is mixed with an oxidant or oxidizing gas to promote pore development (Van den Berg, 2000). Oxidizing gases include oxygen, carbon dioxide, and steam. The process is carried out at a temperature of 800 to 1100 °C. A study by Mahapatra (2009) showed an increase in pore size with an increase in the degree of burning. However, excessive burning may influence the mechanical quality of activated carbon as a result of burning of the walls of activated carbon pores (Mahapatra, 2009; Van den Berg, 2000).

Carbon dioxide and steam react endothermically with the surface of carbon whereas oxygen reacts exothermically (Van den Berg, 2000). The major problem of an exothermic reaction is the burning of the carbon skeleton (Mahapatra, 2009). This affects the development of the porosity of the activated carbon. The use of either carbon dioxide or steam is however, preferred due to their positive effect on the porosity and pore development (Mahapatra, 2009). Activation with carbon dioxide results in the development of micropores with narrow pore size distribution whereas steam produces micropores with wider pore size distribution (Van den Berg, 2000; Mahapatra, 2009).

2.8.2 Characterization of Activated Carbon

Pore System Classification

The pores of activated carbon are classified based on their equivalent diameter (Davidson, 1992). According to Mahapatra (2009) and TIGG Library (2015), the pores diameters are classified as:

- Macropores (greater than 100 nm), they have low contribution to the total internal surface area. They have a significant capability for holding a liquid or liquid retention.
- Mesopores or transitional pores (10-100 nm), contribute to 5% of the total internal surface area. They act as a route for the transportation of the dissolved gold to the micropores
- Micropores (less than 10 nm), contribute to 95% of the total internal surface area. The adsorption of dissolved gold occurs in micropores.

The structure of the activated carbon; surface and internal structure can be analysed using different techniques such as SEM and BET analysis.

Structural Analysis by SEM

Scanning electron microscope (SEM) is a brand of an electron microscope used to conduct inspection of the structure of a sample or specimen. It produces images of high resolution which enables proper analysis of the sample (Mahapatra, 2009). This is done by the production of an electron beam from the electron source which is then accelerated toward the sample by a voltage of a range of 0.2 kV to 30 kV (Orloff, 1997). The reaction between the beam of electrons and sample leads to emission of x-rays, secondary and backscattered electrons which are then collected by a detector. A detector plays a major role in the conversion of x-rays and electrons into a signal which is then displayed into a computer screen (Orloff, 1997; Mahapatra, 2009).

SEM analysis can be done for better understanding of the effect of flotation reagents on the surface and pore structures of activated carbon (Mahapatra, 2009).

BET Analysis

BET stands for Brunauer Emmett Teller. It is an instrument used to determine the specific surface area of a porous solid material (Thomas *et al*., 1996). In addition, it can be used to determine the pore volume and pore area using adsorption and desorption techniques (Mahapatra, 2009). BET analysis is applied in a variety of applications such as medical services, ceramics industries, nanotechnology industries, gold and cement industries (Thomas *et al*., 1996).

In the gold industry, it helps to differentiate between virgin carbon, fouled carbon and regenerated carbon in terms of surface area, pore size and pore volume. Mahapatra (2009) showed the difference in pore volume between virgin carbon (0.454 m^3/g) and regenerated carbon (0.375 m^3/g). Likewise, the study showed the difference in the surface area of virgin and regenerated carbon. Virgin carbon and regenerated carbon were shown to possess the surface

area of 864 m^2/g and 721 m^2/g respectively. This shows that BET analysis is a necessary method to determine the surface area, pore size and pore volume of activated carbon.

2.8.3 Adsorption Mechanism

Various studies (Van den Berg, 2000; Mahapatra, 2009; Gray, 1999; Srithammavut, 2008) have shown a number of proposed mechanisms involved during the adsorption of gold. These mechanisms include:

- Adsorption of $Au(CN)_2^-$ ion.
- Adsorption of the Au(CN) decomposed from $Au(CN)_2^-$.
- Adsorption of the metallic gold reduced from $Au(CN)_2^-$
- Adsorption of $Au(CN)_2^-$ ion in the form of cation

Studies by Davidson (1992) and Vegter (1992) concluded that the process does not involve the reduction of $Au(CN)_2^-$ to metallic state or Au(CN); it only involves the adsorption of $Au(CN)_2^-$ in the form of a cation. Likewise, studies by Adams and Fleming (1989) concluded that the adsorption of gold di-cyanide complex is impossible in the absence of stabilising cations such as potassium, sodium and calcium.

The adsorption mechanism of activated carbon consists of the mass transportation of gold across the solution film surrounding the activated carbon particle, followed by the transportation of gold along the pores of activated carbon by surface diffusion, and then the adsorption reaction onto activated carbon (Salarirad *et al.*, 2011). Although, activated carbon is ideal for adsorption of gold, it has poor selectivity characteristics due to adsorption of dissolved organic and inorganic compounds present in leach solution. The adsorption of unwanted compounds leads to high losses of gold, and lowers the production of gold bullion and thus, company revenue (Kasongo, 2008).

The extent of gold adsorption by activated carbon is determined by the surface area and the presence of a graphitic structure (Burnett, 2001; Mahapatra, 2009). A study by Mahapatra (2009) showed that the adsorption of gold occurred on carbon with a graphitic structure whereas diamond which has no graphitic structure did not adsorb gold. This shows that a graphitic

structure plays a major role during the adsorption of gold. This is due to presence of adsorption sites located at the edge defects in the graphite structure (Mahapatra, 2009; Burnett, 2001).

2.8.4 Gold Adsorption Process

a) Quantity of Gold Adsorbed

The quantity of gold adsorbed by activated carbon is controlled by various factors such as the concentration of gold and unwanted compounds in the solution (Rees, 2000). A study by Salarirad *et al*. (2011) showed that the equation for determining the quantity of gold adsorbed by activated carbon can be expressed as follows:

$$Aa = \frac{Ai - Af}{C} V \quad \text{..... (2)}$$

Where Aa: Quantity of gold adsorbed by activated carbon (mg/g)

C: weight of activated carbon (gram)

V: volume of gold solution (L)

Ai: initial concentration of gold (mg/l)

Af: final concentration of gold after the adsorption (mg/l)

From the equation above, the quantity of gold adsorbed by activated carbon depends on the weight of activated carbon in the circuit/system, concentration of gold before and after the adsorption, and volume of gold solution (Salarirad *et al*., 2011).

b) Adsorption Kinetics

The equation for determining the kinetic activity of gold sorption onto activated carbon was obtained from the Mintek rate equation and is called an empirical kinetic equation (Ross, 1993; La Brooy *et al*., 1984). It can be expressed as follows:

$$\text{Log [Au]} = \log k + n \log t \quad \text{where [Au]} = [\text{Au}]_c^t / [\text{Au}]_s^t \text{ ..... (3)}$$

Where $[\text{Au}]_c$: quantity of gold adsorbed by activated carbon after time t (mg/l)

$[Au]_s$: quantity of gold remained in the solution after time t (mg/l)

k : kinetic rate constant expressed in a unit per hour

t : time for adsorption (hour)

n : empirical constant dependent on activated carbon (slope of line)

Virgin activated carbon has a k-value of about 1100/hr (La Brooy *et al*., 1984). Therefore, a higher value of k indicates that there is higher adsorption rate. Fouling of the activated carbon tends to lower the value of k (Mahapatra, 2009). In addition, the use of distilled water tends to lower the value of k. A study by Fisher (2000) showed that the use of distilled water led to a decrease in the value of k from 1200 to 640/hr. The study by Salarirad *et al*. (2011) concluded that there is a need to increase the residence time or concentration of activated carbon if the value of k decreases below 50/hr.

c) Adsorption Efficiency

The study by Ndi Nsami *et al*. (2013) showed that the adsorption efficiency or percentage formula involved during the adsorption of gold by activated carbon can be expressed as follows:

$$\%R = \frac{Ai - Af}{Ai}\ 100 \quad \ldots\ldots\ldots\

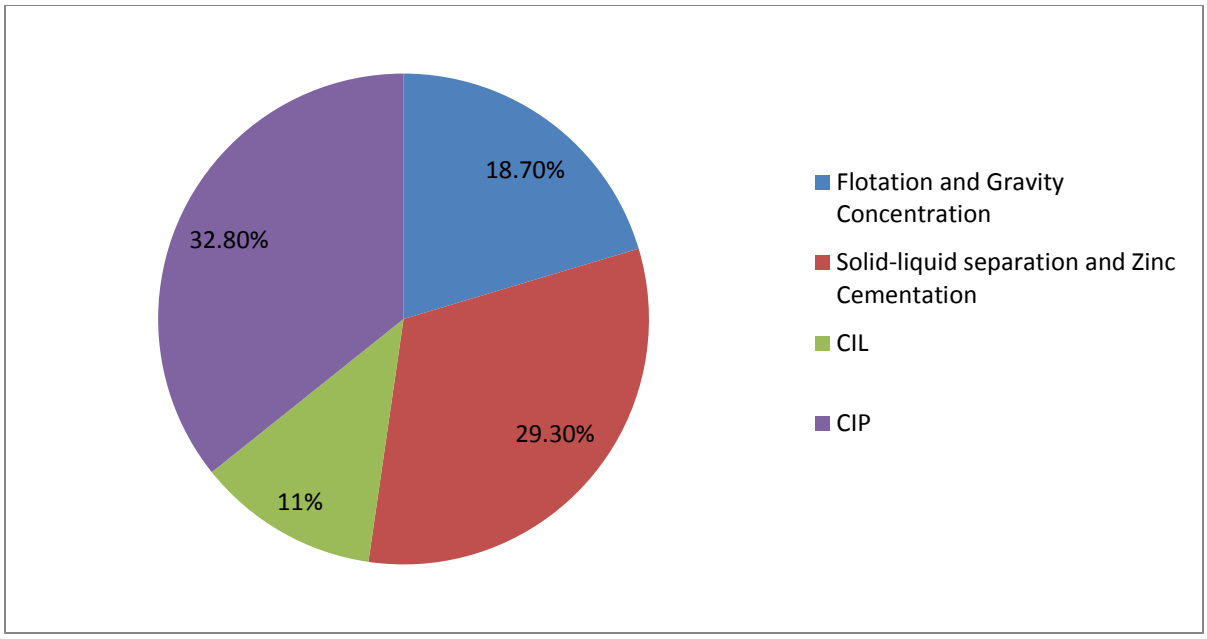


Figure 2.4: Gold recovery methods in the world (Srithammavut, 2008)

2.9.1 Carbon in Leach Process

In the CIL process, leaching and adsorption of gold take place concurrently within the same tank (Srithammavut, 2008). Fresh or regenerated activated carbon is added to adsorb gold as soon as it is dissolved by the chemical cyanide. This method is mostly applied to preg-robbing ores, and ores that contain large quantity of organic matter (de Andrade Lima, 2007). Preg-robbing ores tend to compete with activated carbon for gold adsorption whereas organic matter results in fouling of the activated carbon. This lowers the amount of gold adsorbed onto activated carbon.

The advantage of this method is on the reduction of capital cost (Srithammavut, 2008). There is no separate tank construction for the leaching and also for the adsorption of gold. In addition, there is no requirement for a solid liquid separation step thus saving on capital and operational costs (Marsden and House, 2006). Although the process has low operating costs, it however, requires high capital cost and larger carbon inventory (Srithammavut, 2008; Marsden and House, 1992). This is due to the construction of larger agitated tanks to give sufficient residence time for leaching and adsorption. Moreover, it needs a huge carbon inventory as a result of carbon losses. Carbon losses occur due to the attrition process as a result of carbon movement from one tank to another (Marsden and House, 2006; Srithammavut, 2008). The movement leads to collision

between carbon particles and hence lowers the size of carbon. Fine carbon leads to the loss of carbon and adsorbed gold as a result of its ability to pass through the inter-stage screen (Marsden and House, 2006).

2.9.2 Carbon in Pulp Process

CIP is used when the ore does not contain a large quantity of organic and preg-robbing material (Marsden and House, 2006). It consists of two main stages, which are gold cyanide leaching followed by gold adsorption. The process does not require the construction of bigger agitated tanks like CIL (Srithammavut, 2008). However, it cannot treat complex ores such as those with preg-robbing characteristics (Srithammavut, 2008; Marsden and House, 1992, 2006).

2.10 Factors Affecting Gold Adsorption by Activated Carbon

There are several factors which affect the adsorption of gold by the activated carbon. These factors include mixing efficiency, carbon type and size, gold and cyanide concentration, time, solution pH and temperature, ionic strength and carbon fouling (Marsden and House, 2006).

2.10.1 Time

Although the adsorption of gold takes place within the first 8 hours, it is however necessary to increase the contact time to achieve higher loading (Srithammavut, 2008). Salarirad *et al.* (2010) showed that an increase in time from 2 hours to 8 hours led to an increase in gold adsorption from 3000 g/t to 4000 g/t. Furthermore, the studies showed the effect of time on the loading capacity of non-fouled activated carbon. Non-fouled carbon achieved a loading capacity of 3870 ppm within the first 8 hours while fouled activated carbon achieved the same loading capacity in 15 hours. This shows that an increase in residence time plays a significant role in the gold adsorption process especially with regards to fouled carbon.

2.10.2 Temperature

The adsorption rate of gold increases with an increase in temperature; however the loading capacity decreases with an increase in temperature (Metalliferous Mining- Process, 2010). The increase in temperature results in an increase in the mass transfer of gold to the surface of activated carbon. A decrease in the loading capacity is due to the exothermic nature of the reaction during the adsorption process (Davidson, 1992; Coetzee, 1996). Davidson (1992) showed that an increase in temperature from 16 to 40 °C led to a decrease in loading capacity from 35 to 16 kg/t. Furthermore, the increase in temperature tends to influence the adsorption equilibrium (McDougall *et al*., 1980; Vegter *et al*., 1996). Studies by Vegter *et al*. (1996) showed the plating of elemental gold as a result of an increase in temperature. Loaded carbon (more than 350 mg/l) was kept in contact with solution at a temperature of 70 and 90 °C. The solution turned yellowish indicating that there was a plating of elemental gold. The study concluded that the occurrence of plating was due to the instability of gold di-cyanide complex as a result of high temperature, high gold concentration and the absence of free cyanide.

2.10.3 Oxygen Concentration

Oxygen plays a major role during the oxidation of active sites of the activated carbon and hence equilibrium loading (Coetzee, 1996). Davidson (1992) showed an increase in kinetic activity from 28 to 36/hr as a result of the replacement of air by pure oxygen. This shows that the presence of oxygen promotes the loading of gold onto the activated carbon.

2.10.4 Carbon Particle Size

The adsorption rate increases with a decrease in the size of carbon particles (Mahapatra, 2009). Fine carbon possesses a larger pore diameter and a smaller internal surface area than coarse carbon. The study by Mensah-Biney *et al*. (1996) showed that a decrease of carbon size from +10 mesh to -20 mesh results in an increase in the adsorption rate from 0.27 to 1.24 $(kg/t)^{-1}$ h r^{-1}.

2.10.5 Cyanide Concentration

Cyanide has been shown to promote the selectivity of carbon during the adsorption of gold (Gray, 1999). Davidson (1992) showed that an increase in cyanide concentration results in an increase in the loading capacity of activated carbon. The increase of cyanide concentration from 0 to 200 g/t led to the increase of loading capacity from 0.50 kg/t to 0.78 kg/t respectively. However, the study also showed that high cyanide levels results in an increase in the competition for active sites between gold and free cyanide ions. It was concluded therefore, that it is necessary to optimise cyanide concentration in the circuit to prevent the adsorption of free cyanide ions.

Another study by Mahapatra (2009) showed the role of cyanide in minimising the fouling effect of xanthate on activated carbon. Xanthate influences the adsorption rate and loading capacity of gold onto the activated carbon. The xanthate molecules form a layer on the external surface of the activated carbon, and further adsorb on and block the pores of the activated carbon (Salarirad *et al*., 2011, Petersen *et al*., 1997; Mahapatra, 2009). This results in the reduced adsorption and loading capacity of the carbon. However, the presence of free cyanide in a solution has been shown to lower the fouling effect of the xanthate (Mahapatra, 2009).

2.10.6 pH

A decrease in pH has been shown to promote the adsorption of gold onto activated carbon due to the decrease in the competition for active sites on the activated carbon between gold and hydroxide ions (Gray, 1999).

The study by Davidson (1992) showed that an increase in pH from 3 to 9 led to a decrease in the loading capacity from 55 to 20 mg/g. However, most of the adsorption processes are carried out at pH of 10.5 to 11 for safety purposes such as to prevent the formation of hydrogen cyanide gas (Gray, 1999).

2.10.7 Gold Concentration

A high concentration of gold in the leach solution results in an increase in the adsorption rate and loading capacity (Gray, 1999). This is due to an increase in mass transfer rate of gold to the surface of activated carbon. Davidson (1992) showed that an increase in gold concentration in a leach solution from 0.10 to 10 ppm resulted in an increase in the loading capacity from 10 to 60 kg/t.

2.10.8 Slurry Density

An increase in slurry density leads to a decrease in the adsorption rate and loading capacity (Gray, 1999). This is due to a temporary or permanent blinding of the exposed pores and surfaces of activated carbon by the ore particles. In addition, an increase in pulp density tends to increase viscosity and makes mixing to become more complex (Jones *et al*., 1989). Therefore, it is recommended to operate at a percent solid of 40% to 45% to prevent the sinking of activated carbon and to minimise gold losses.

2.10.9 Carbon Concentration

An increase in carbon concentration has been shown to be one of the solutions to overcoming carbon fouling caused by organic and inorganic compounds. Mahapatra (2009) showed a decrease in the solution gold concentration as a result of an increase in carbon concentration. The gold concentration in solution was reduced from 2.092 to 0.067 mg/ml as a result of an increase in carbon concentration from 0.1 to 0.5 gm/l.

2.10.10 Ionic Strength

The increase in the content of free ions leads to an increase in the adsorption rate and loading capacity of gold (McDougall *et al*, 1980). McDougall *et al.* (1980) showed the effect of an increase in the concentration of $CaCl_2$. Their testwork was carried out in the absence of and also at 0.067 M $CaCl_2$ and residual gold was found to be 140 mg/l and 90 mg/l respectively. Davidson (1992) found that the adsorption of gold depends on the concentration and nature of the spectator

cations in the adsorption media. The study concluded that the adsorption of gold is impossible in the absence of stabilising cations such as sodium, potassium and calcium. This indicates that the adsorption of gold onto activated carbon takes place in the presence of cations and the reaction is given by:

$$pAu(CN)_{2}^{-}{}_{(aq)} + N^{p+}{}_{(aq)} = N(Au(CN)_{2})_{p}\,(carbon)$$

Where N = Ca, Mg, Na and K, and p is a valency of N

Davidson (1992) also showed that an increase in the adsorption of gold from 3 mg/g to about 12 mg/g occurred as a result of replacement of deionized water with tap water. Furthermore, a study by Gray (1999) concluded that the adsorption of gold is enhanced by the charge of the cation and not the size of a cation. The charge of cations was expressed in the series below:

$$Ca^{2+} > Mg^{2+} > H^{+} > Li^{+} > Na^{+} > K^{+}$$

The adsorption of gold is better in the presence of calcium ions compared to magnesium, lithium, and sodium as a result of the high ionic strength of the solution (McDougall *et al.,* 1980).

2.10.11 Fouling

Fouling is the adsorption of unwanted compounds on the surface or pores of activated carbon. This occurs due to poor selectivity of activated carbon (Salarirad *et al*., 2011). Fouling influences carbon activity and results in an increase in carbon inventory and residence time, and an increased number of adsorption stages. The adsorption of foulants is caused by the presence of polar and non-polar molecules present in unwanted compounds (Davidson, 1992).

The introduction of hydrophobic particles (non-polar) in water develops a structured water cage. Hydrophobic particles tend to destroy the structure of the hydrogen bond network and, as a

result, a cage is formed by the hydrogen bond around particles. Hydrophobic particles tend to run away from the water structure resulting in the adsorption onto the surface of hydrophobic particle. In addition, there is attraction of polar molecules to the surface of activated carbon. Attraction is achieved by the use of the hydrogen bond which makes the surface of carbon hydrophilic (Davidson, 1992). This influences the adsorption of gold onto carbon particles due to the less availability of active site on the activated carbon.

There are two types of unwanted compounds that influence the adsorption rate and loading capacity in CIL/CIP circuits. These are inorganic and organic compounds.

a) Inorganic Compounds

Most inorganic compounds are associated with gold deposits and hence are present during the leaching of gold. These includes mercury, zinc, iron, nickel, calcium, magnesium, copper, and cobalt (Marsden and House, 2006; Mahapatra, 2009). Some are present due to the addition of reagents in the circuit (Mahapatra, 2009). The addition of lime in the circuit as a pH modifier results in the introduction of calcium as an inorganic foulant. The study by Davidson (1992) showed that an increase in calcium concentration to above 50 kg/t led to a decrease in the adsorption of gold by activated carbon.

Inorganic compounds are leached together with gold as a result of their presence in the agitation leaching tank. The leached metal ions tend to influence the adsorption of gold by activated carbon. This is due to their competition with gold complex ions for the available active sites of activated carbon (Burnett, 2001). However, a study by Fleming and Nicol (1984) concluded that copper and iron have a high influence compared to all other metal complexes as a result of their high concentration in most of the gold ore deposits. Copper has the ability to oxidise free cyanide in the solution, and its loading ability onto activated carbon is controlled by the free cyanide concentration and pH levels. $Cu(CN)_2^-$ is formed at low free cyanide concentration and low pH, and it tends to compete with gold for adsorption onto activated carbon. $Cu(CN)_3^-$ and $Cu(CN)_4^-$ are formed at a high free cyanide concentration and high pH, and have a negligible effect on gold adsorption onto activated carbon (Burnett, 2001; Davidson, 1992). The adsorption of $Cu(CN)_2^-$ is therefore higher than that of $Cu(CN)_3^-$ and $Cu(CN)_4^-$ due to its high solubility (Davidson,

1992). This indicates that the dissolution of gold by cyanide should take place at high pH and high free cyanide concentration. However, the use of high free cyanide concentration and high pH can cause a negative effect on the adsorption of gold onto activated carbon especially during CIL processes (Ross, 1993; Marsden and House, 2006, Davidson, 1992). They tend to lower the number of active sites that were made for the adsorption of gold.

b) Organic Compounds

Organic compounds include oil, fats, greases, ethanol, methanol, acetone, flotation reagents, vegetation products and dewatering reagents (La Brooy *et al.*, 1986). Their presence is due to the addition of flotation and dewatering reagents in the circuit. Moreover, it is due to the addition or spillage of oil during the maintenance of processing equipment (Salarirad *et al*., 2011). Adsorption of organic compounds occur by mechanisms which are primarily a balance between hydrogen bonding in the leach solution and Van der Waals forces of attraction between the surface of carbon and organic compounds (Davidson, 1992).

The studies by Mahapatra (2009) and Salarirad *et al*. (2011) concluded that frothers and collectors are the main organic compounds that cause a serious deactivation of the activated carbon. Table 2.1 summarizes examples of oils, frothers and collectors that cause a serious deactivation on the activated carbon.

Table 2.1: Categories of organic compounds and their examples

	Type of Agent	Examples
1	Collectors	Sodium Ethyl Xanthate, Potassium Amyl Xanthate, Potassium Ethyl Xanthate, Aero 238 Promoter,
2	Frothers	X- 133 frother, Methyl Isobutyl Carbinol, Teric 401, Dowfroth 200
3	Oils	Multigrade lubricant, Diesel

Frothers are heteropolar compounds added to stabilize the loaded bubble and prevent it from bursting. The use of frothers such as methyl isobutyl carbinol (MIBC), teric frothing agents and x-133 have been shown to be a major problem during the adsorption of gold onto activated carbon. A study by Salarirad *et al*. (2011) has shown than an increase in frother concentration (from 0 to about 25 mg/l) leads to a decrease in the kinetic activity of activated carbon (500/hour to 300/hour). This was attributed to film formation on the surface of the activated carbon which then prevents mass transfer of gold onto activated carbon. It was however, concluded that, the fouling effect of MIBC on activated carbon is negligible at low concentration (below 15mg/l). In addition, the study by Fisher (2000) showed the fouling effect of teric frothing agents and MIBC on the kinetic activity of activated carbon. The fouling effect of the teric frothing agent was found to be more critical compared to MIBC. This was due to the effect of molecular weight, teric frothing reagent has a larger molecular weight compared to MIBC. The effect of molecular weight was also studied by Traube (1891) and led to the introduction of Traube's rule (Fisher, 2000; Mahapatra, 2009). This rule predicts that, the longer the chain molecule in the homologous series, the stronger is the adsorption onto activated carbon. The study concluded that frothers affect the adsorption rate and not the loading capacity of gold onto activated carbon. However, the study by Mahapatra (2009) showed that there is blocking of activated carbon pores by frother molecules which affect both the adsorption rate and loading capacity. The testwork involved the use of a solution containing oreprep x-133 as a frother, distilled water, sodium cyanide, and 0.1 mg/ml of activated carbon. The adsorption efficiency of frother onto activated carbon was found to be 98.48%.

Xanthate is the most common collector used in the flotation circuit due to its longer hydrocarbon chain which influences the degree of hydrophobicity on sulphide minerals (Wills, 2006). The presence of xanthate during CIP/CIL, affects the adsorption rate of gold onto activated carbon. A study by Salarirad *et al*. (2011) showed a decrease in the kinetic activity of activated carbon (500 to 100 per hour) with an increase in xanthate concentration (0 to 80 mg/L). The study concluded that xanthate affects the adsorption rate and not the loading capacity of gold onto activated carbon. In addition, xanthate with longer hydrocarbon chains (larger molecular weight) was seen to have more effect on the kinetic activity than on the loading capacity. Fisher (2000) showed a decrease in the kinetic activity of activated carbon from 280/hour to 40/hour due to the use of ethyl against amyl xanthate. However, a study by Mahapatra (2009) noted that there is blocking

of activated carbon pores by xanthate molecules which affect both the adsorption rate and loading capacity. Testwork involved the use of a solution containing potassium amyl xanthate, distilled water, sodium cyanide, and 0.1 mg/ml of activated carbon. The adsorption efficiency of xanthate onto activated carbon was 99.78%. On the other hand, a study by Petersen *et al*. (1997) showed that large molecules of flotation reagents did not cause the blocking of pores. The study however showed that the effect was only due to the competitive effect between gold di-cyanide complex and organic compounds on the active sites of activated carbon surface. The conclusion was that low organic loading on the carbon surface affects only the rate of gold adsorption whereas high organic loading affects both the adsorption rate and the loading capacity of gold di-cyanide complex onto activated carbon. The study also showed the competition of adsorption (expressed in terms of competition factor) between sodium ethyl xanthate (SEX) and gold, and potassium amyl xanthate (PAX) and gold. The competition factor between SEX and gold was 1.264 whereas for PAX and gold was 1.236. The difference was due to the larger molecular weight of SEX compared to the lower moleclar weight of PAX (160.28 against 144 g/mol).

2.11 Techniques for the Removal of Flotation Reagents

Pre-treatment of flotation concentrate before CIL and CIP processes is necessary to overcome the effect of flotation reagents (Salarirad *et al*., 2010). Studies by Bilston *et al*. (1990) and Fisher (2000) also concluded that pre-treatment of flotation concentrate is necessary to remove or dislocate flotation reagents present in the mineral surface before further processing.

Pre-treatment may be done by processes such as the use of sodium sulphite, filtration, oxidation and a combination of both filtration and oxidation. In addition, pre-treatment can be done by roasting which leads to the burning of all flotation molecules present in the concentrate.

2.11.1 Application of Sodium Sulphite

Sodium sulphite plays a major role in either desorbing or decomposing flotation reagents from the mineral surface (Jones and Woodcock, 1988). Bilston *et al*. (1990) showed a decrease in xanthate concentration from 56 ppm to 15 ppm due to the addition of 3.65 kg/t of sodium sulphite. The decrease in xanthate concentration resulted in an increase in dissolution efficiency

from 24.5% to 26%. It was concluded that an increase in sodium sulphite concentration and the addition of a catalyst such as copper might improve the extraction of gold.

2.11.2 Oxidation

Oxidation has been shown to displace flotation reagents from the mineral surface. Studies by Bilston *et al*. (1990) showed a decrease in xanthate concentration from 56 ppm to 13 ppm due to the addition of 12.5 l/t of hydrogen peroxide. The decrease in xanthate concentration resulted in an increase in the dissolution efficiency from 24.5% to 56%. This suggests that hydrogen peroxide plays a significant role in lowering the fouling effect of xanthate on activated carbon.

2.11.3 Filtration and Oxidation

A combination of both filtration and oxidation has been shown to result in great improvements in gold extraction. Bilston *et al*. (1990) showed that an increase in gold extraction from 24.5% to 65% and 88% in a cyanidation period of 24 hours and 48 hours respectively was due to the treatment of solids by lime and tap water for 5 minutes followed by the addition of hydrogen peroxide. This shows that a combination of filtration and oxidation may dislocate flotation reagents present in the mineral surface.

2.11.4 Roasting

Roasting is a pre-treatment method that can be used to decompose flotation reagents present on the mineral surface (Fisher, 2000). The decomposition of flotation reagents such as SEX react to produce gaseous ethylene and carbonyl sulphide, and solid residue of sodium hydrogen sulphide (Fisher, 2000). In addition, studies by Dunn *et al*. (1997) reported the production of dixanthogens, elemental sulphur, metallic sulphides, thiols and dialkyl xanthates.

Study by BHP engineers (1989) proposed a process flowsheet for the application of roasting in the decomposition of flotation reagents present on the mineral surface of gold concentrates at the Mouteh gold processing plant. However, the use of roasting was subsequently replaced by ultra-

fine grinding due to the significant impact of the roasting process on the environment. Mouteh gold processing plant (after the replacement of roasting with ultra-fine grinding) was closed for a month after the operation as a result of the high extent of organic fouling caused by flotation reagents (Salarirad *et al*., 2011). This suggests that the use of roasting could have prevented the closure of the plant.

2.12 Regeneration of Activated Carbon

The main aim of regeneration is to restore carbon to the level of virgin carbon (Marsden and House, 2006; Ross, 1993). This tends to lower the operating costs. The fouled activated carbon can be reactivated by two methods, acid washing and thermal reactivation (Mahapatra, 2009).

2.12.1 Acid Washing

The acid washing process helps to remove the adsorbed inorganic compounds and allow the sites to be available for the adsorption of gold. However, the removal is not as effective as compared to the thermal reactivation process (described below). This is because removal of compounds happens only on the surface of activated carbon and not inside the pores (Mahapatra, 2009).

Acid washing can be carried out using a hot process or cold process. Hot process uses hot hydrochloric acid whereas cold process uses cold hydrochloric acid (Mahapatra, 2009; Davidson, 1992). The study by Mahapatra (2009) showed that the significant improvement of carbon activity observed from hot acid washing was due to the removal of large concentration of calcium, copper, magnesium, and iron as compared to cold acid washing.

2.12.2 Thermal Reactivation

Whilst acid washing is applied before the removal of gold from the activated carbon, thermal reactivation is applied after the removal of gold. The process increases the activity and loading capacity of activated carbon. The process is carried out in a kiln called a regeneration kiln and involves the use of heat and tumbling to regenerate fouled carbon (Davidson, 1992).

Overall Summary

Although flotation is a necessary pre-cyanidation step required to recover gold which is finely disseminated in sulphide minerals, the flotation reagents can cause significant deactivation of activated carbon. This is due to lack of selectivity of activated carbon. The deactivation is more significant with an increase in the concentration of flotation reagents and with the use of larger molecular weight compounds such as xanthate.

Flotation reagents tend to form a layer on the external surface of activated carbon (Salarirad *et al*., 2010; 2011). This layer affects the adsorption rate and not the loading capacity of gold onto the activated carbon. Other studies (Fisher, 2000; Mahapatra, 2009) have shown that the flotation reagents have the potential to affect both the adsorption rate and loading capacity of gold onto activated carbon. Flotation reagents are adsorbed on the surface of activated carbon and not onto the pore of activated carbon. This is due to inaccessibility of molecules of flotation reagents onto the pore of activated carbon. High adsorption influences the adsorption rate and the loading capacity of gold onto the activated carbon. Likewise, flotation reagents are loaded onto the pores of activated carbon, and, influence the adsorption rate and the loading capacity of gold onto the activated carbon. They influence the adsorption rate and loading capacity as a result of blocking of the activated carbon pore. Blocking of the activated carbon pores inhibit mass transfer rate of gold onto the activated carbon.

Furthermore, carbon fouling is caused by high concentration of free cyanide and high pH. This shows that the use of high cyanide levels results in an increase in the competition for active sites of the activated carbon between gold and free cyanide ions. Likewise, the use of pH above 11 results in an increase in the competition for active sites of the activated carbon between gold and calcium ions.

The deactivation of activated carbon results in high residual gold in the circuit, poor adsorption rate of gold onto activated carbon, and poor loading of gold onto activated carbon

CHAPTER THREE

MATERIALS AND METHODS

3.1 Introduction

The purpose of the experiments was to study the fouling effect of PAX (collector) and MIBC (frother) on the adsorption rate and loading capacity of gold by activated carbon. Experiments were based on the comparison of non-fouled and fouled activated carbon using a single CIP stage. The following procedures were conducted to achieve the objectives:

- Preparation of gold standard solution
- Fouling of the activated carbon
- Activity tests of both fouled and non-fouled activated carbons
- Solution sample analysis
- Carbon sample analysis

The description of facilities and testwork procedures are discussed in the following sections.

3.2 Materials

Equipment

The laboratory batch testwork equipment were an overhead stirrer and a bottle roll machine equipped with a 2 litre beaker and 1 litre GL 45 glass bottle respectively. In addition, a sieve of 45 microns, measuring cylinder, spatula, syringes, analytical balance for weighing of the activated carbon and AuCN particles, and a Metrohm 744 pH meter to measure the level of acidity and alkalinity were used.

Reagents

Reagents were sourced from Sigma-Aldrich, Senmin and Merck companies. Sodium cyanide and lime were obtained from the chemical stores at the University of the Witwatersrand. Gold (I) cyanide and frother (MIBC) were provided from Sigma-Aldrich whereas the collector (PAX) and virgin activated carbon were supplied from Senmin and Merck respectively. All the reagents were of analytical grade.

3.3 Experimental Procedures

Stage 1: Preparation of $Au(CN)_2$ Standard Solutions

The process involved the preparation of 100 ppm of $Au(CN)_2$ standard solution using AuCN particles. This is defined by the reaction below:

$$Au(CN) + CN \rightarrow Au(CN)_2$$

1.5 g of lime was added into a bottle containing 1000 mL distilled water in order to attain the required pH of 10. This was followed by the addition of 0.1 g of AuCN, and lastly the addition of 0.3 g of NaCN to dissolve AuCN solid particles. The mixture was agitated in an overhead stirrer for 24 hours at a speed of 80 rpm.

Stage 2: Fouling of Activated Carbon

This process was divided into two steps. Step 1 involved the fouling of activated carbon using different concentration of individual PAX and MIBC while step 2 involved the fouling of the carbon using the combination of both PAX and MIBC. The procedure was as follows:

Step 1

Fouling of activated carbon involved the use of different concentration of individual PAX and MIBC, and a combination of both reagents (PAX plus MIBC). For individual concentration, 0.1g of PAX was added into a bottle containing 1000 mL distilled water, giving an equivalent of 100ppm of PAX. A total of 3 bottles of 100ppm each of PAX were prepared and in each bottle a

specific amount of activated carbon was added. The amounts of activated carbon added were 1 g, 5 g and 10 g. This was followed by the addition of 1.5 g of lime and 250 ppm of NaCN. The mixtures were placed on a bottle roll machine and agitated at 50 rpm. After 24 hours, the fouled activated carbon was removed from the mixtures using a sieve of 45 microns and added into a gold solution to determine the adsorption efficiency.

The procedure outlined above was repeated for individual preparation of 300 ppm of PAX, 100 ppm of MIBC and 300 ppm of MIBC. The same procedure was also followed in the preparation of a mixture with a combination of both PAX and MIBC (100 ppm of PAX plus 300 ppm of MIBC and 300 ppm of PAX plus 100 ppm of MIBC).

Step 2

0.1 g of PAX and 0.3 g of MIBC were added into a bottle containing 1000 mL distilled water to give an equivalent of 100 ppm of PAX and 300 ppm of MIBC. A total of three bottles were prepared and in each bottle a specific amount of the activated carbon was added. The amounts of activated carbon added were 1 g, 5 g and 10 g. The process was followed by the addition of lime and 250 ppm of NaCN. The mixture was placed on a bottle roll machine and agitated at 50 rpm. After 24 hours, the fouled activated carbon was then removed from the mixture by using a sieve of 45 microns and added into a gold solution to determine the adsorption efficiency.

The same procedure was repeated for a mixture of 0.3 g of PAX and 0.1 g of MIBC. This was aimed to give an equivalent of 100 ppm of MIBC and 300 ppm of PAX.

Stage 3 Activity Tests

Carbon activity test was based on a single CIP stage, and involved the use of fouled and non-fouled activated carbon for comparison using a typical CIP set up. 1 g of activated carbon (1 g of fresh carbon and fouled activated carbon) were added into a bottle which contained 130.84 mL distilled water, 23.96 mL of 100 ppm of $Au(CN)_2$ and 200 ppm of NaCN. It was followed by agitation on a bottle roll machine at 50 rpm so as to allow for the adsorption of gold present in solution. The time for adsorption was set to be 12 hours. A sample solution was collected at desired time intervals of 0.5, 1, 2, 3, 4, 6, 8, 10 and 12 hours. The aim was to determine the

amount of gold remaining in the solution, adsorption capacity of activated carbon, and the kinetics of adsorption. Table 3.1 shows the operating parameters used in the testwork.

Table 3.1: Operating parameters tested

Process condition/parameter	Value
Residence time during the dissolution of AuCN particles by NaCN	24 Hours
Carbon: PAX ratio (g : ppm)	1:100, 5:100, 10:100, 1:300, 5:300, and 10:300
Carbon: MIBC ratio (g : ppm)	1:100, 5:100, 10:100, 1:300, 5:300, and 10:300
Carbon: PAX: MIBC ratio (g : ppm : ppm)	1:100:300, 5:100:300, 10:100:300, 1:300:100, 5:300:100, and 10:300:100
Adsorption time	12 hours total
Lime added	pH was maintained at 10

Stage 4 Analysis of Solution Samples

Samples were analyzed using atomic absorption spectroscopy (200 Series AA) found at Richard Ward building at the University of the Witwatersrand. The aim was to determine gold concentration in pregnant solution (head sample), adsorption efficiency (loading and adsorption rate) onto activated carbon, and fouling effect of flotation reagents on activated carbon.

Stage 5 Analysis of Carbon Samples

Carbon samples were analyzed using Scanning Electron Microscope (SEM) found at Richard Ward building at the University of the Witwatersrand. The aim was to understand the pore

structure of the activated carbon. In addition, the carbon was analyzed using energy dispersive spectroscopy (EDS) in order to determine the elemental composition of the activated carbon. Likewise, the carbon was analyzed using Brunauer Emmett Teller (BET) in order to determine the surface area, pore volume, and pore size.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 BET Analysis for Non-fouled and Fouled Activated Carbon

BET analysis was done on non-fouled and fouled activated carbon for better understanding of the effect of flotation reagents on the average surface area, pore volume and pore size of carbon. The comparison of non-fouled and fouled activated carbon is shown in Table 4.1.

Table 4.1: BET analysis for non-fouled and fouled activated carbon

Sample ID	surface area (m^2/g)	Pore volume (cm^3/g)	Pore size (nm)
Non-fouled carbon	956.4919	0.495083	2.56489
300 ppm of MIBC fouled carbon	649.5746	0.349817	2.39799
300 ppm of PAX fouled carbon	568.6793	0.309975	2.42428

The results in Table 4.1 confirm that flotation reagents cause a decrease in the average surface area and pore volume of the fouled carbon. The reduction in the pore size was however, not very significant. Non-fouled activated carbon shows an average surface area of 956.4919 m^2/g, pore volume of 0.495083 cm^3/g, and pore size of 2.56489 nm. This is quite large compared to 649.5746 m^2/g, 0.349817 cm^3/g and 2.39799 nm for MIBC fouled carbon and 568.6793 m^2/g, 0.309975 cm^3/g and 2.42428 nm for PAX fouled carbon. The decrease in average surface area, pore volume and pore size may be explained by either adsorption or loading of flotation reagents onto activated carbon (Petersen *et al*., 1997, Ross, 1993).

4.2 SEM Images of Non-fouled and Fouled Activated Carbon

SEM analysis was done on non-fouled and fouled activated carbon for better understanding of the effect of flotation reagents on the surface and pore structures of activated carbon. The results are shown in Figure 4.1, 4.2 and 4.3. It can be seen that the surface and pores of non-fouled carbon are free from particles compared to the surfaces and pores of fouled carbon. This indicates that there might be a loading or deposition of flotation reagents onto activated carbon.

SEM images support the study by La Brooy *et al*. (1987), in which, ultraviolet absorbance showed the loading of larger chains of xanthate molecules onto the activated carbon. La Brooy *et al*. (1987), indicated that the loading of larger chains of xanthate molecules leads to severe deactivation of the activated carbon. To support this assumption of loading of flotation reagents onto the activated carbon, SEM-EDS analysis was conducted to determine elemental surface composition of the activated carbon. The depth of analysis for EDS machine ranges between 1-3 microns. Table 4.2 shows the elemental composition of the non-fouled and fouled activated carbon.

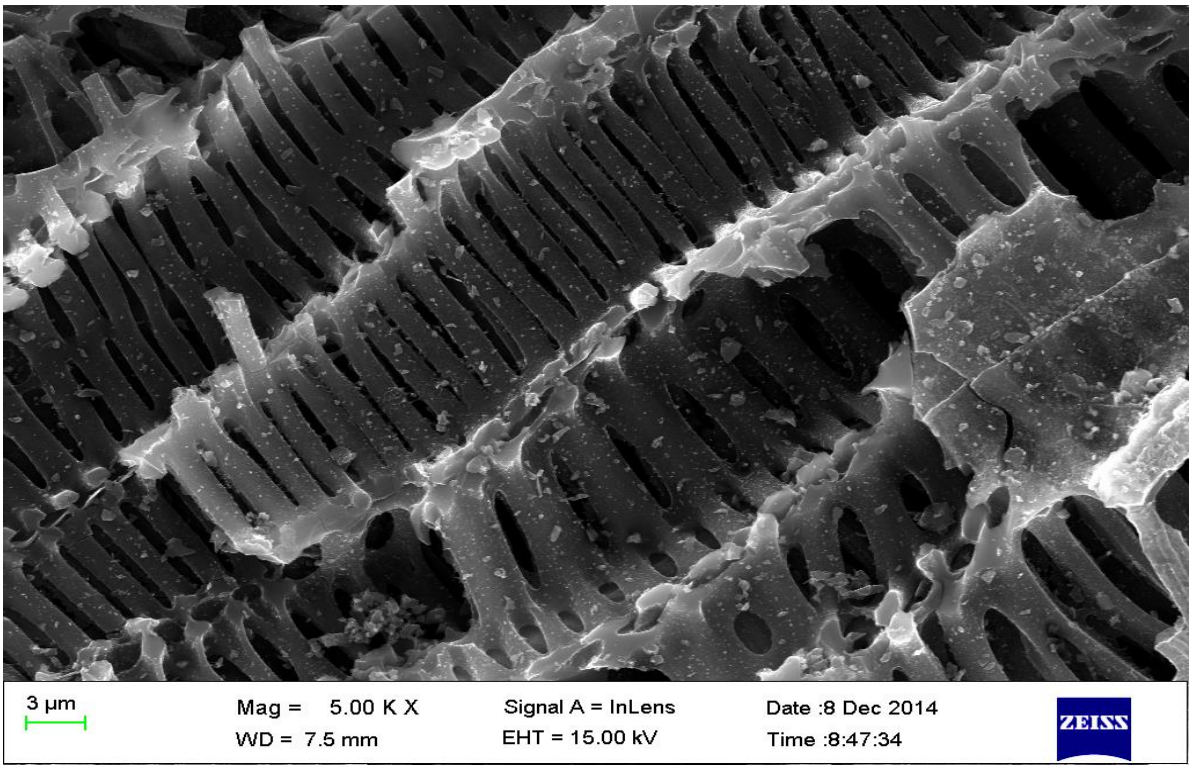


Figure 4.1: SEM image of non-fouled activated carbon at magnification of 5000x

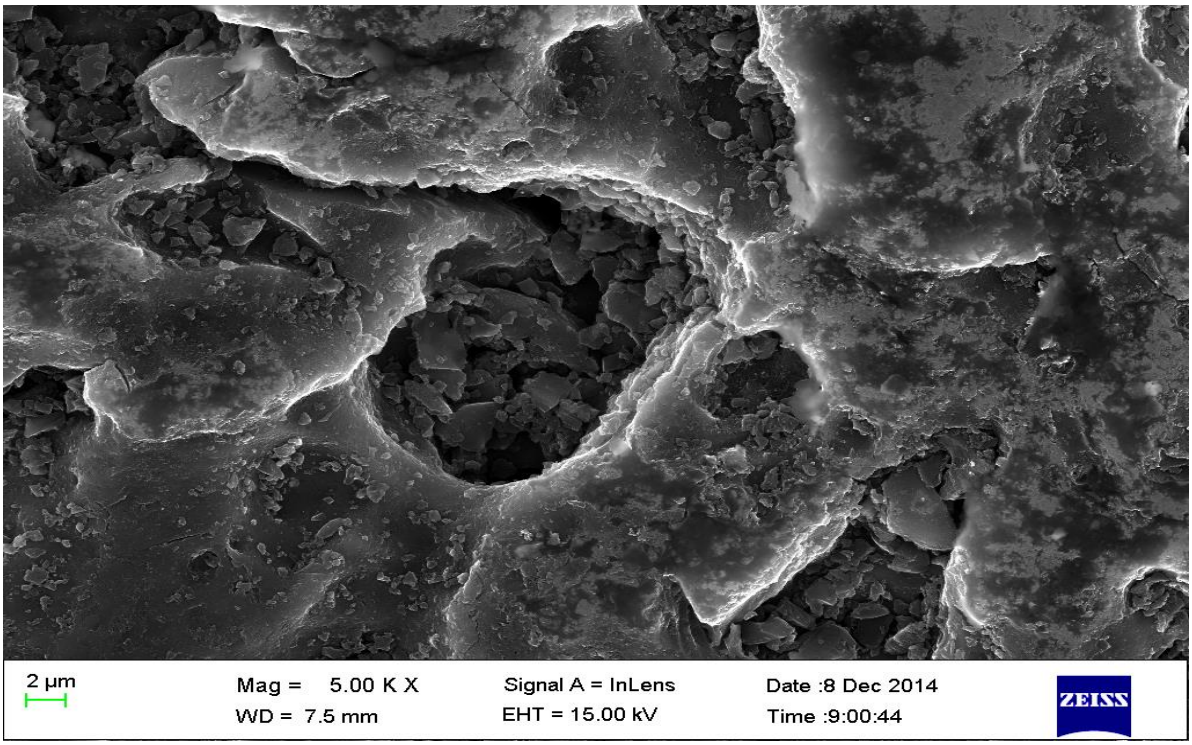


Figure 4.2: SEM image of 300 ppm MIBC fouled activated carbon at magnification of 5000x

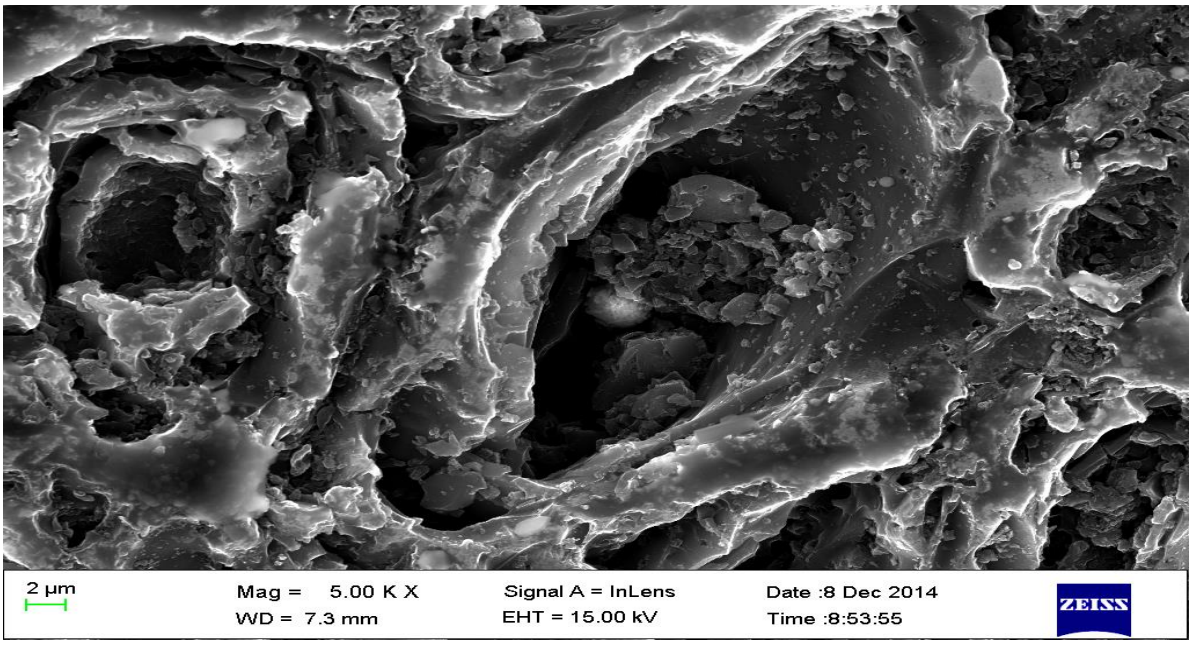


Figure 4.3: SEM image of 300 ppm PAX fouled activated carbon at magnification of 5000x

Table 4.2: SEM-EDS surface structural analysis for non-fouled and fouled activated carbon

Element	Non- fouled carbon		Fouled carbon by 300 ppm of MIBC		Fouled carbon by 300 ppm of PAX	
	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
C	94.18	95.6	90.00	92.78	89.11	93.43
O	5.71	4.35	8.67	6.71	5.92	4.66
Na	0	0	0.20	0.11	0.16	0.09
Mg	0	0	0.19	0.10	0.09	0.05
Si	0	0	0.12	0.05	0	0
S	0.110	0.05	0	0	3.58	1.41
K	0	0	0.20	0.06	0.73	0.24
Ca	0	0	0.62	0.19	0.40	0.13
Total	100		100		100	

From Table 4.2, it can be seen that the effect caused by PAX on the surface structure of activated carbon is more significant compared to the effect caused by MIBC. This is noted through the high composition of elements (excluding carbon and oxygen) on the surface structure of the PAX fouled activated carbon than the MIBC fouled carbon. These elements are core components of the flotation reagents (PAX and MIBC). They contribute to about 4.96 % by weight on PAX fouled carbon and 1.33 % by weight on MIBC fouled carbon. Moreover, BET analysis also showed a significant decrease in the surface area of PAX compared to the MIBC fouled carbon.

The elemental composition of the pores was analyzed using EDS. Pore elemental composition of non-fouled and fouled activated carbon is shown in Table 4.3. It can be seen that the effect caused by PAX on the pores of activated carbon is more significant as compared to the effect

caused by MIBC. This is noted through a higher elemental composition on the pores of the PAX fouled activated carbon. They contribute to about 8.36 % by weight on PAX fouled carbon and only 3.18 % by weight on MIBC fouled carbon. In addition, BET analysis showed a significant decrease in pore volume and pore size of PAX fouled carbon than MIBC fouled carbon. The elemental composition was high on the pores of fouled carbon than on the surface structure of fouled carbon. This may be explained by the adsorption mechanism of activated carbon. The adsorption mechanism of activated carbon consists of the mass transportation of molecules across the solution film surrounding the activated carbon particle, followed by the transportation of molecules along the pores of activated carbon by surface diffusion, and then the adsorption reaction onto activated carbon (Salarirad *et al*., 2011). This indicates that more molecules will be loaded or deposited into the pores of activated carbon. Moreover, the high composition of elements on PAX fouled carbon may be explained by a larger molecular weight of PAX (Mahapatra, 2009; Fisher, 2000). The adsorption of larger molecular weight compounds (longer chain molecules) is much stronger than that of smaller molecular weight compounds (Fisher, 2000).

Table 4.3: SEM-EDS pore elemental composition analysis of non-fouled carbon and fouled activated carbon

Element	Non-fouled carbon		Fouled carbon by 300 ppm of MIBC		Fouled carbon by 300 ppm of PAX	
	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
C	94.18	95.6	89.28	92.92	77.42	89.57
O	5.71	4.35	7.54	5.89	2.21	1.92
Na	0	0	0.32	0.17	0.17	0.10
Mg	0	0	0.54	0.28	0.23	0.13
Si	0	0	0	0	0.59	0.29
K	0	0	0.87	0.28	5.32	1.89
S	0.110	0.05	0	0	14.06	6.09
Ca	0	0	1.46	0.45	0	0
Totals	100		100		100	

SEM and EDS analysis shows that there are particles on the surface structure and pores of fouled activated carbon that come from PAX and MIBC. These particles show an elemental composition that includes sodium, magnesium, silicon, calcium, sulphur and potassium. Thus, it can be concluded that the loading capacity and adsorption rate of gold onto the activated carbon will not be similar for non-fouled and fouled activated carbon.

4.3 The Fouling Effect of Flotation Reagents

4.3.1 The Effect of PAX on Gold Adsorption

PAX is a type of sulphydryl collector consisting of anionic polar and uncharged non polar hydrocarbon groups (Wills, 2006). It is mostly used in the flotation of gold ores, associated with sulphide minerals due to its high selectivity compared to other sulphydryl collectors (Avotins *et al*., 1994). Moreover, the sulphydryl collectors have a longer hydrocarbon chain which increases the degree of hydrophobicity of a sulphide mineral (Wills, 2006; Gupta, 2003).

Figure 4.4 and 4.5 illustrates the residual gold content during the carbon activity test for fresh carbon and carbon fouled by 100 ppm and 300 ppm of PAX respectively. From the figures, it can be observed that non-fouled carbon (fresh carbon) has a residual gold of 0.11 ppm after 12 hours of contact. This is very small compared to the residual gold arising from the carbon fouled by PAX. Activated carbon fouled by 100 ppm of PAX shows a residual gold of 6.86 ppm, 4.16 ppm and 2.84 ppm for 1 g, 5 g and 10 g of activated carbon respectively. The increase of PAX concentration to 300 ppm results in an increase in the residual gold to 12.84 ppm, 7.94 ppm and 4.73 ppm for 1 g, 5 g and 10 g of carbon respectively.

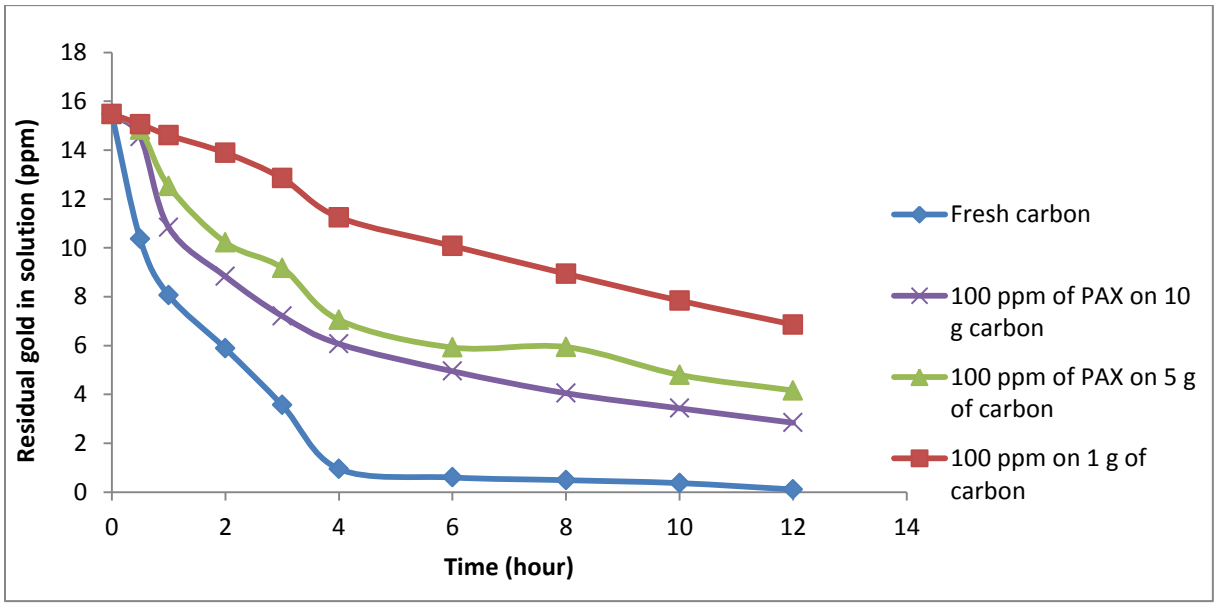


Figure 4.4: Residual gold for fresh carbon and carbon fouled by 100 ppm of PAX

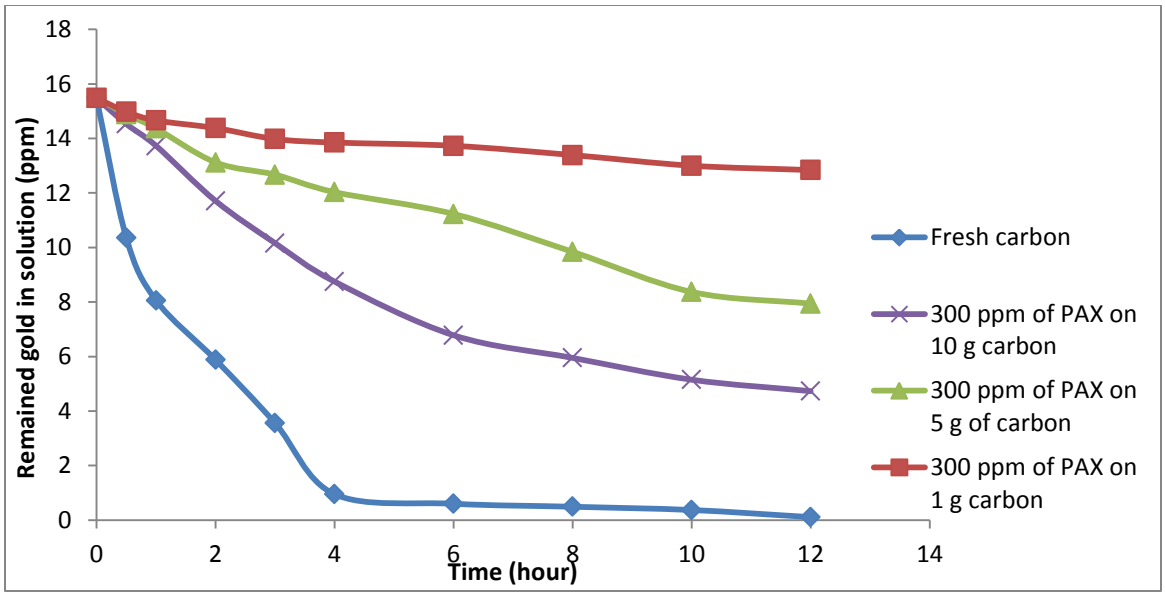


Figure 4.5: Residual gold for fresh carbon and carbon fouled by 300 ppm of PAX

A study by Salarirad *et al*. (2011) showed that the equation for determining the quantity of gold adsorbed by activated carbon can be expressed as follows:

$$Aa = \frac{Ai - Af}{C} V \quad \text{..... (2)}$$

Where Aa: Quantity of gold adsorbed by activated carbon (mg/g)

C: weight of activated carbon (gram)

V: volume of gold solution (L)

Ai: initial concentration of gold (mg/l)

Af: final concentration of gold after the adsorption (mg/l)

Equation 2 was used to determine the amount of gold adsorbed onto the carbon. Figure 4.6 and 4.7 show the loading capacity of fresh carbon and carbon fouled by 100 ppm and 300 ppm of PAX respectively.

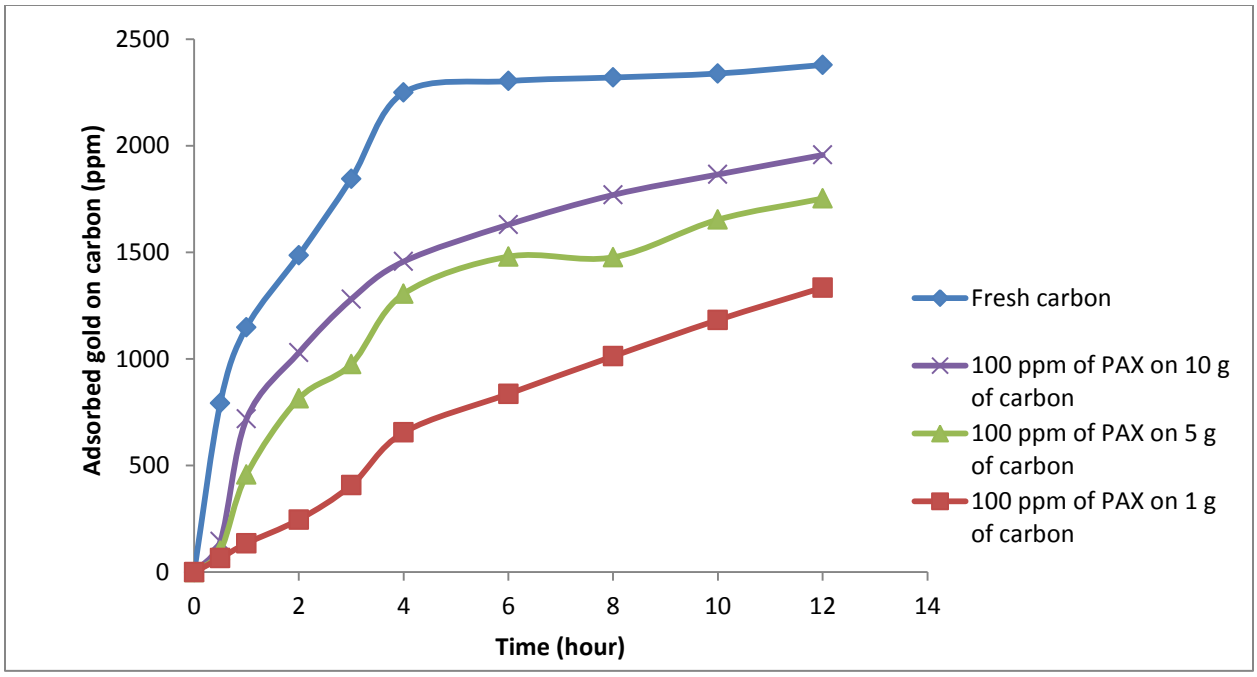


Figure 4.6: Loading capacity of fresh carbon and carbon fouled by 100 ppm of PAX

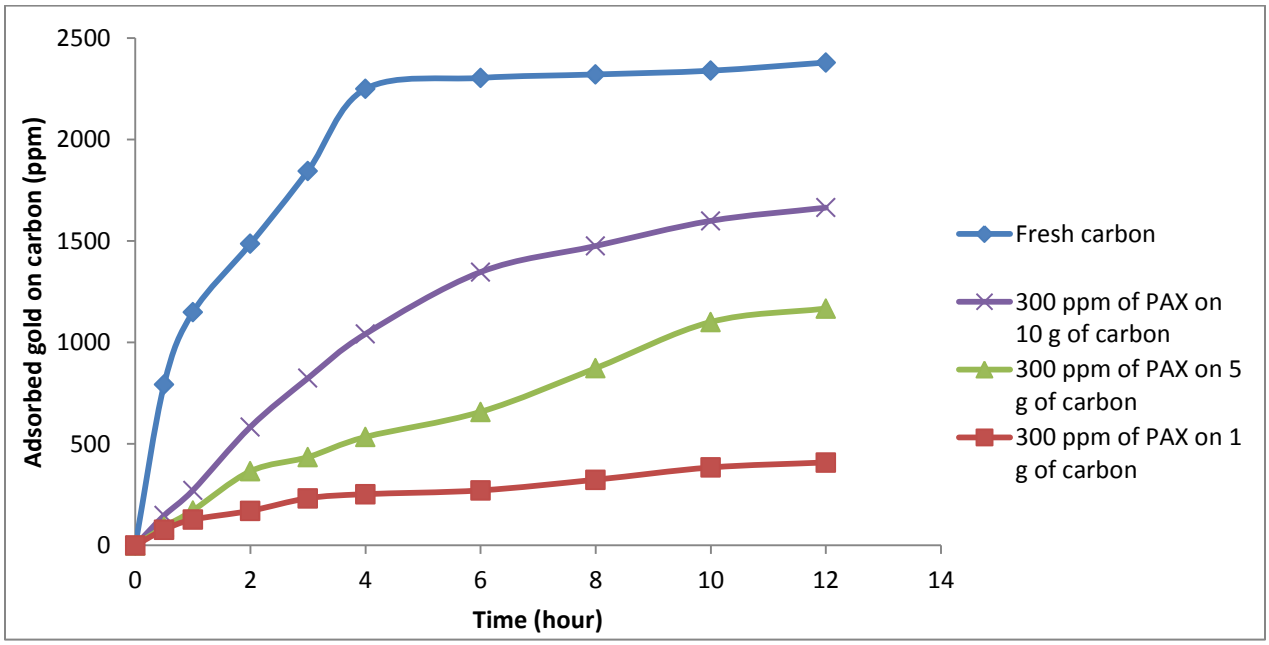


Figure 4.7: Loading capacity of fresh carbon and carbon fouled by 300 ppm of PAX

From Figure 4.6 and 4.7, it can be observed that non-fouled activated carbon achieved a loading capacity of 2379 ppm whereas carbon fouled by 100 ppm of PAX achieved a loading capacity of 1335 ppm, 1752 ppm and 1957 ppm for 1 g, 5 g and 10 g of activated carbon respectively. The

increase of PAX concentration to 300 ppm showed a significant deactivation of the activated carbon. This is shown in the decreased loading capacity of 409 ppm, 1167 ppm and 1664 ppm for 1 g, 5 g and 10 g of activated carbon respectively. However, the increase of carbon concentration increased the loading of gold onto the activated carbon. An increase of carbon concentration from 1 g to 10 g led to an increase in the loading capacity from 1335 ppm to 1957 ppm for 100 ppm of PAX and 409 ppm to 1664 ppm for 300 ppm of PAX.

4.3.2 Effect of PAX on the Adsorption Kinetics

The efficiency of the adsorption circuit is determined by the kinetic activity (k-value) of gold sorption onto the activated carbon. The presence of PAX in the adsorption circuit results in a poor kinetic activity. The major consequences of poor kinetic activity are an increase in the residence time, carbon inventory and size of the adsorption stages (La Brooy *et al*., 1984). Therefore, it is necessary to have higher kinetic activity to increase the adsorption rate and lower the operating cost.

The equation for determining the kinetic activity of gold sorption onto activated carbon was obtained from the Mintek rate equation and is called an empirical kinetic equation (Ross, 1993; La Brooy *et al*., 1984). It can be expressed as follows:

$$\text{Log } [Au] = \log k + n \log t \quad \text{where } [Au] = [Au]_c^t / [Au]_s^t \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

Where $[Au]_c$: quantity of gold adsorbed by activated carbon after time t (mg/l)

$[Au]_s$: quantity of gold remained in the solution after time t (mg/l)

k : kinetic rate constant expressed in a unit per hour

t : time for adsorption (hour)

n : empirical constant dependent on activated carbon (slope of line)

From equation 3, it can be concluded that the plot of log $(\Delta[Au]^t_c / [Au]^t_s)$ against log t is a straight line and the kinetic activity (k-value) can easily be obtained from the log $(\Delta[Au]^t_c / [Au]^t_s)$ intercept. The study by Salarirad *et al*. (2010) concluded that the equation is linear within the first 4 hours of the adsorption.

Figure 4.8 and 4.9 show the k-values for gold loading onto fresh carbon and carbon fouled by 100 ppm and 300 ppm of PAX respectively.

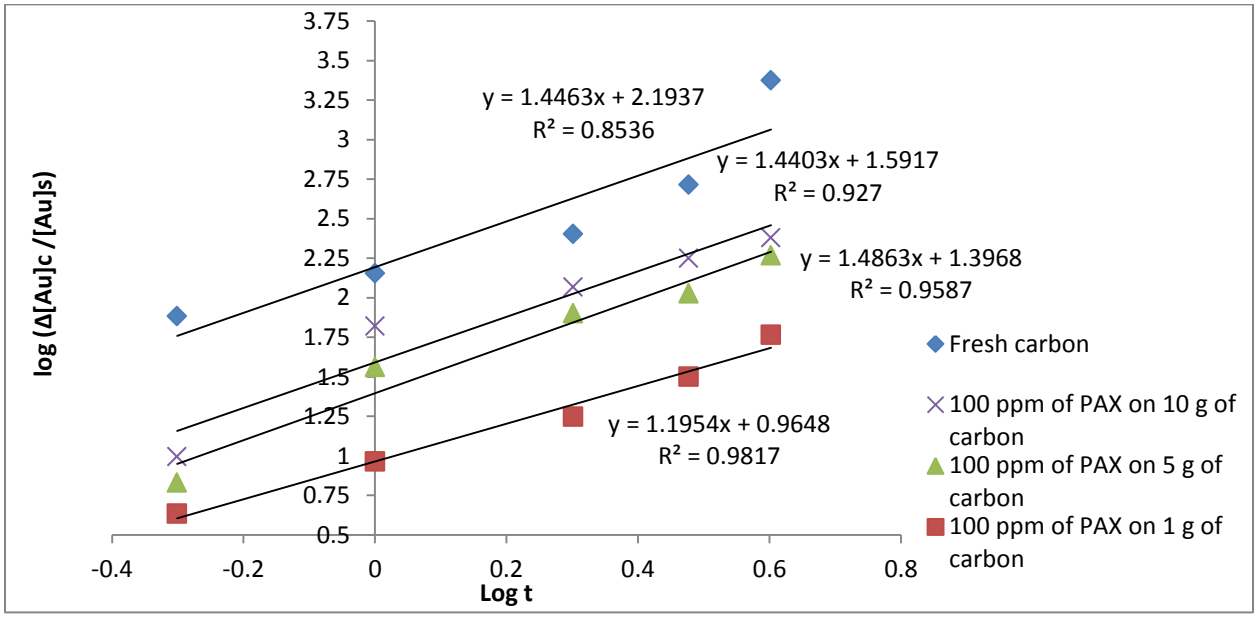


Figure 4.8: Plot of log ($\Delta[Au]_c^t$ /$[Au]_s^t$) vs log t for fresh carbon and carbon fouled by 100 ppm of PAX.

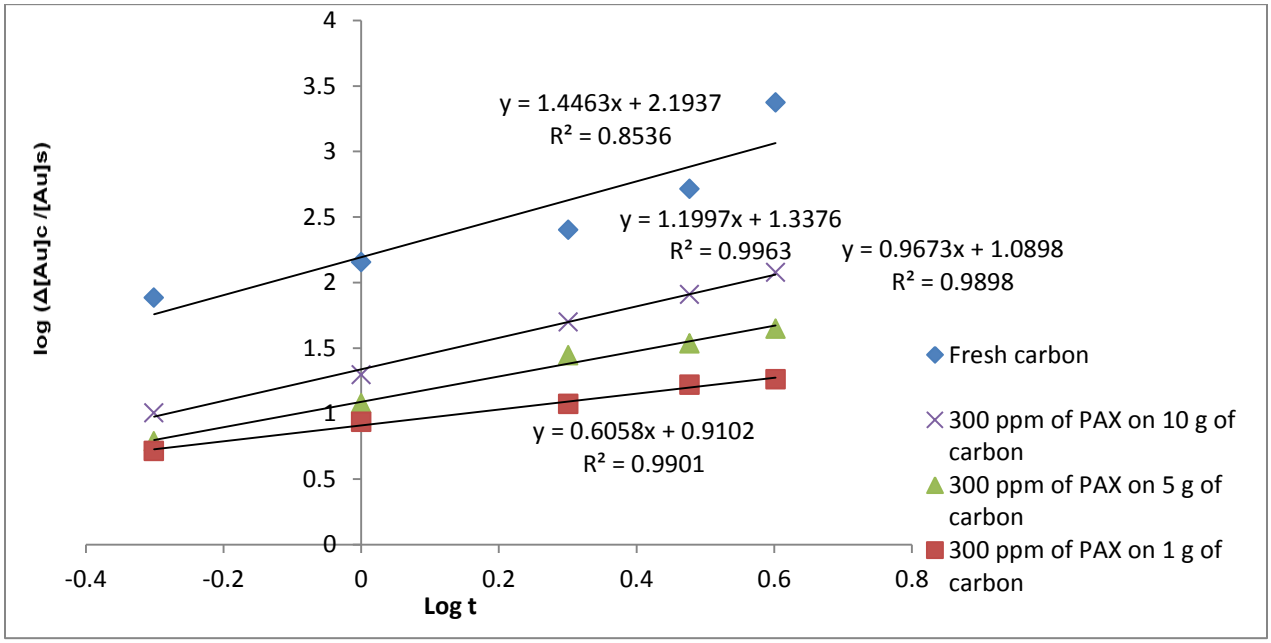


Figure 4.9: Plot of log ($\Delta[Au]_c^t$ /$[Au]_s^t$) vs log t for fresh carbon and carbon fouled by 300 ppm of PAX.

Table 4.4 gives a summary of the k-values for gold loading onto non-fouled carbon and PAX fouled carbon as calculated from the information in the Figure 4.8 and 4.9.

Table 4.4: k-values for gold loading onto non-fouled activated carbon and PAX fouled activated carbon.

	Non-fouled carbon 1g	Fouled carbon by 100 ppm of PAX			Fouled carbon by 300 ppm of PAX		
		1g	5g	10g	1g	5g	10g
K –value ($h^{-1)}$	156	9	25	39	9	12	22

Non-fouled activated carbon shows a high k-value compared to fouled activated carbon. The increase of PAX concentration from 0 to 100 ppm led to a decrease of the k-value from 156 hr^{-1} to 9 hr^{-1}, 25 hr^{-1} and 39 hr^{-1} for 1 g, 5 g and 10 g of activated carbon respectively. Likewise, the increase of PAX concentration to 300 ppm led to a decrease of the k-value to 9 hr^{-1}, 12 hr^{-1} and 22 hr^{-1} for 1 g, 5 g and 10 g of activated carbon respectively. It can be seen that an increase in the carbon concentration resulted in an increase in the k-value. The increase in the carbon concentration from 1 g to 10 g led to an increase of the k-value from 9 hr^{-1} to 39 hr^{-1} for 100 ppm of PAX and 9 hr^{-1} to 22 hr^{-1} for 300 ppm of PAX.

4.3.3 Effect of MIBC on Gold Adsorption

MIBC is a type of frother mostly used in the flotation of gold ores associated with sulphide minerals. MIBC has the same structure as PAX due to the presence of both polar and non-polar groups which means partly water repellent and partly water soluble. MIBC is added in the circuit to prevent the bursting of the bubble and to help stabilize the loaded bubble (Wills, 2006).

MIBC causes similar problems as those resulting from the addition of PAX. Figure 4.10 and 4.11 illustrates the residual gold during the carbon activity test for fresh carbon and carbon fouled by

100 ppm and 300 ppm of MIBC respectively. From the figures, it can be observed that non-fouled carbon has residual gold of 0.11 ppm after 12 hours of adsorption. This is very small compared to the residual gold from the activated carbon fouled by MIBC. Activated carbon fouled by 100 ppm of MIBC shows a residual gold content of 4.98 ppm, 3.85 ppm and 1.07 ppm for 1 g, 5 g and 10 g of activated carbon respectively. The increase in MIBC concentration results in an increase in the residual gold. An increase in MIBC concentration to 300 ppm results in an increase in residual gold to 7.48 ppm, 5.12 ppm and 3.74 ppm for 1 g, 5 g and 10 g of activated carbon respectively. However, the increase in carbon concentration lowers the fouling effect of MIBC. The increase in carbon concentration from 1 g to 10 g led to a decrease in the residual gold from 4.98 ppm to 1.07 ppm (percentage change of 78.5%) for 100 ppm of MIBC, and 7.48 to 3.74 ppm (percentage change of 50%) for 300 ppm of MIBC.

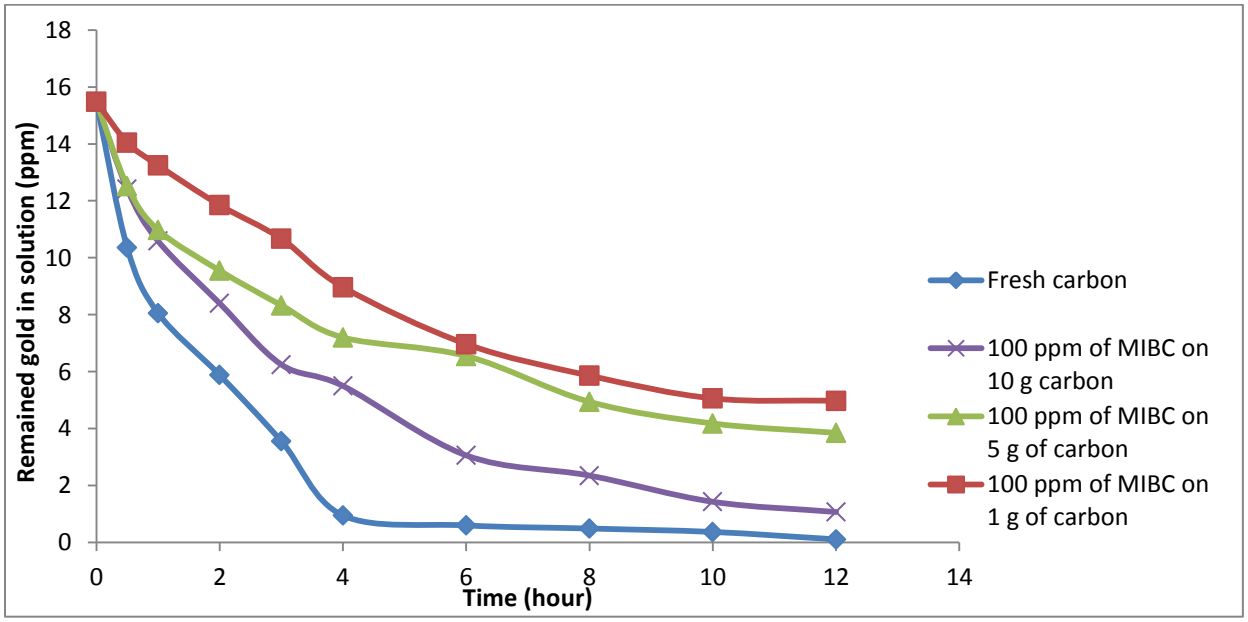


Figure 4.10: Residual gold for fresh carbon and carbon fouled by 100 ppm of MIBC

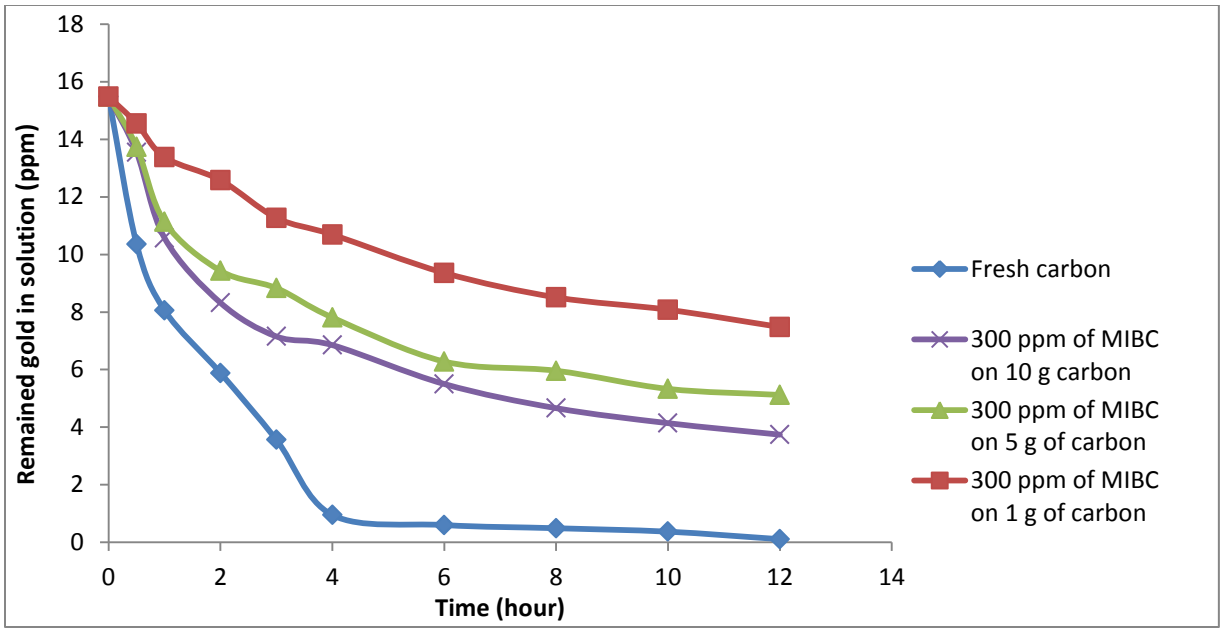


Figure 4.11: Residual gold for fresh carbon and carbon fouled by 300 ppm of MIBC

Equation 2 was used to determine the amount of gold adsorbed onto the activated carbon.

Figure 4.12 and 4.13 show the loading capacity of non-fouled carbon and carbon fouled by 100 ppm and 300 ppm of MIBC respectively.

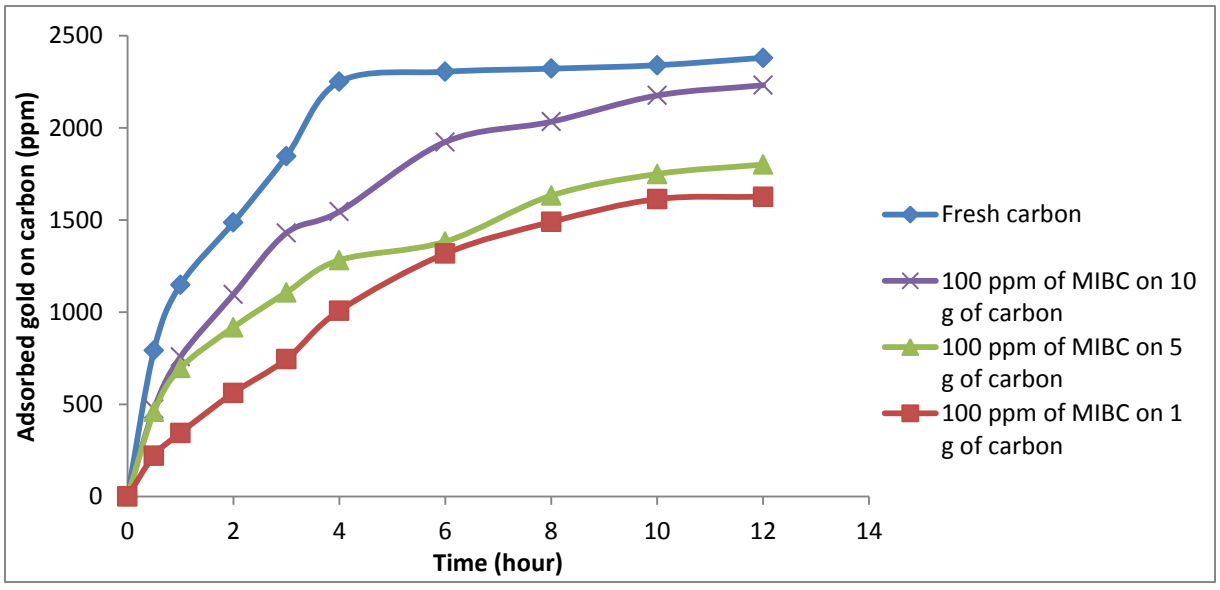


Figure 4.12: Loading capacity of fresh carbon and carbon fouled by 100 ppm of MIBC

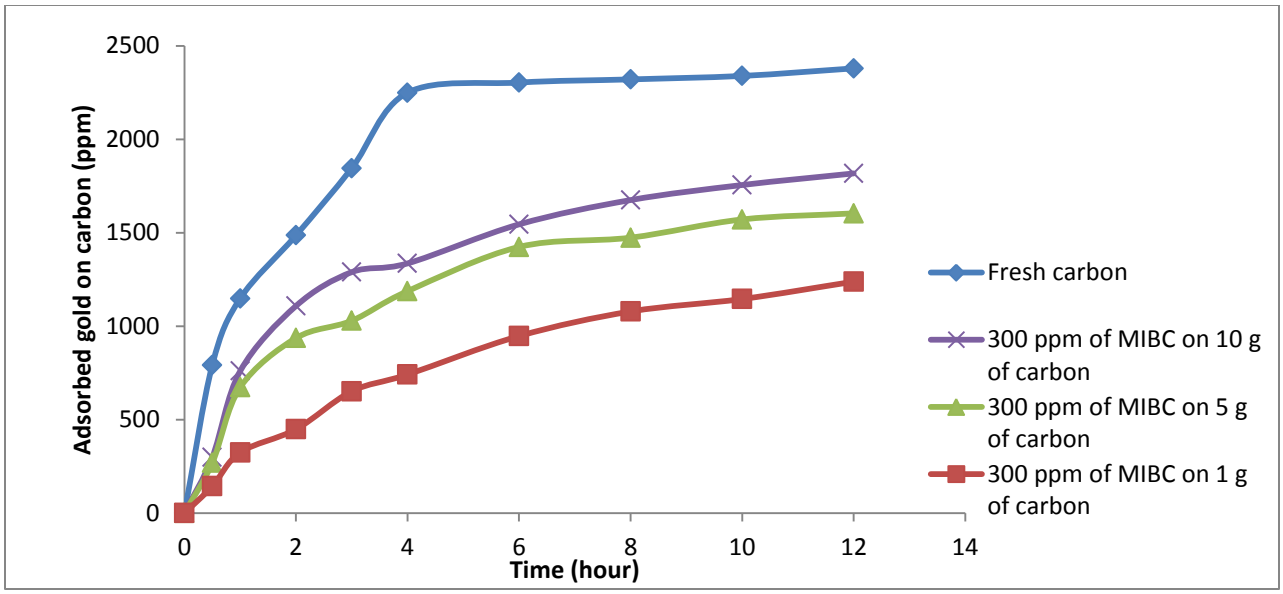


Figure 4.13: Loading capacity of fresh carbon and carbon fouled by 300 ppm of MIBC

It can be observed that non-fouled activated carbon achieved a loading capacity of 2379 ppm whereas carbon fouled by 100 ppm of MIBC achieved a loading capacity of 1625 ppm, 1800 ppm and 2231 ppm for 1 g, 5 g and 10 g of activated carbon respectively. An increase in MIBC concentration to 300 ppm further showed a significant deactivation of activated carbon. This consequently led to a decrease in the loading capacity of the activated carbon to 1238 ppm, 1604 ppm and 1817 ppm for 1 g, 5 g and 10 g of activated carbon respectively. The increase in carbon concentration however, leads to an increase in the loading capacity of activated carbon. The increase in carbon concentration from 1 g to 10 g led to an increase in the loading capacity from 1625 ppm to 2231 ppm for 100 ppm of MIBC and 1238 ppm to 1817 ppm for 300 ppm of MIBC.

4.3.4 Effect of MIBC on the Adsorption Kinetics

The presence of MIBC in the adsorption circuit causes a significant deactivation of the activated carbon.

Equation 3 was used to determine the kinetic activity of gold loading onto activated carbon. Figure 4.14 and 4.15 illustrate the k-values for the loading rate of gold onto fresh carbon and carbon fouled by 100 ppm and 300 ppm of MIBC respectively.

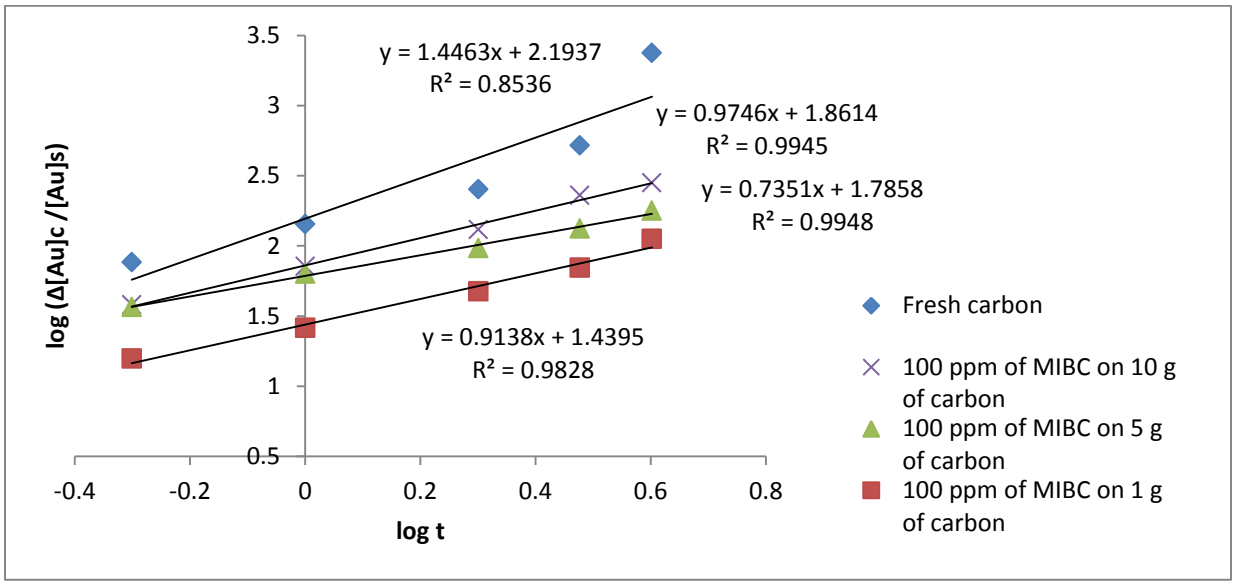


Figure 4.14: Plot of log ($\Delta[Au]_c^t$ /$[Au]_s^t$) vs log t for fresh carbon and carbon fouled by 100 ppm of MIBC

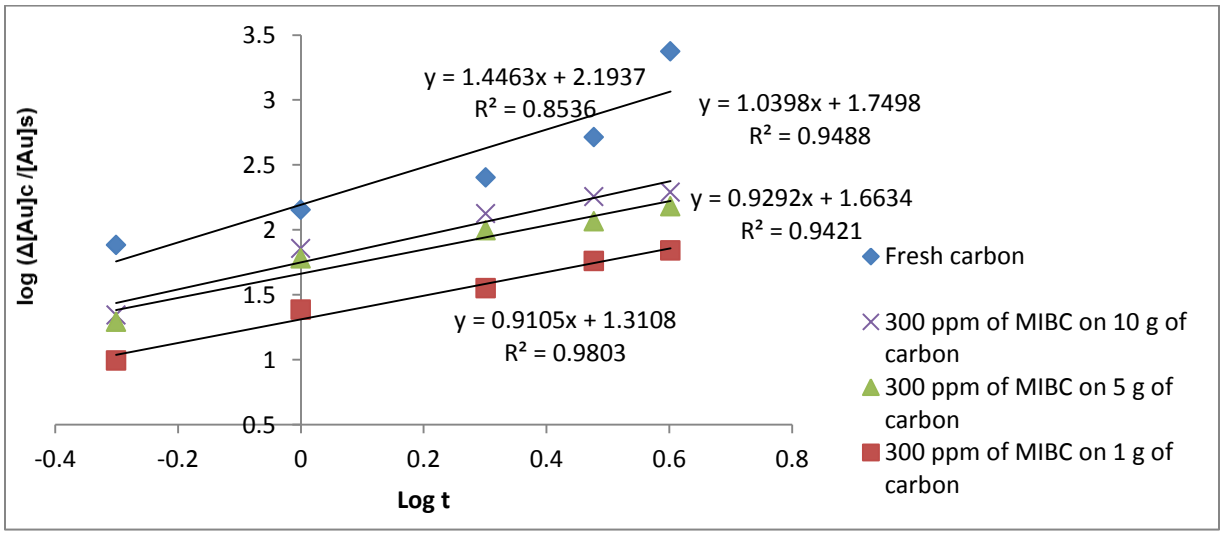


Figure 4.15: Plot of log ($\Delta[Au]_c^t$ /$[Au]_s^t$) vs log t for fresh carbon and carbon fouled by 300 ppm of MIBC

Table 4.5 gives a summary of the k-values for the loading rate of gold onto non-fouled carbon and MIBC fouled carbon as calculated from the information in Figure 4.14 and 4.15.

Table 4.5: k-values for the loading rate of gold onto non-fouled carbon and MIBC fouled activated carbon

	Non-fouled carbon 1g	Fouled carbon by 100 ppm of MIBC			Fouled carbon by 300 ppm of MIBC		
		1g	5g	10g	1g	5g	10g
K –value (h^{-1})	156	28	61	73	20	46	56

Non-fouled activated carbon shows a higher k-value compared to fouled activated carbon. The increase of MIBC concentration from 0 to 100 ppm led to a decrease in the k-value from 156 hr^{-1} to 28 hr^{-1}, 61 hr^{-1} and 73 hr^{-1} for 1 g, 5 g and 10 g of activated carbon respectively. Likewise, the increase of MIBC concentration to 300 ppm led to a decrease in the k-value to 20 hr^{-1}, 46 hr^{-1} and 56 hr^{-1} for 1 g, 5 g and 10 g of activated carbon respectively. However, the increase in the carbon concentration tends to increase the k-value for gold loading onto activated carbon. The increase in carbon concentration from 1 g to 10 g led to an increase in the k-value from 28 hr^{-1} to 73 hr^{-1} for 100 ppm of MIBC and 20 hr^{-1} to 56 hr^{-1} for 300 ppm of MIBC.

4.3.5 Effect of a Mixture of PAX and MIBC on the Loading Capacity of Activated Carbon

The fouling effect caused by a combination of PAX and MIBC on the adsorption of gold onto the activated carbon was evaluated through batch experiments. Figure 4.16 and 4.17 illustrate the residual gold for fresh carbon and carbon fouled by 100 ppm of MIBC and 300 ppm of PAX, and 300 ppm of MIBC and 100 ppm of PAX respectively. From the figures, it can be observed that non-fouled carbon has a residual gold of 0.11 ppm after 12 hours of adsorption. This is very small compared to the residual gold resulting from the carbon fouled by a combination of PAX

and MIBC. Carbon fouled by a combination of 100 ppm of MIBC and 300 ppm of PAX shows a residual gold of 13.4 ppm, 10.6 ppm and 5.1 ppm for 1 g, 5 g and 10 g of carbon respectively. The effect was much higher compared to carbon fouled by a combination of 100 ppm of PAX and 300 ppm of MIBC. In this case, residual gold was found to be 11.62 ppm, 4.91 ppm and 3.19 ppm for 1 g, 5 g and 10 g of carbon respectively.

The mixture of 100 ppm of MIBC and 300 ppm of PAX achieved a high residual gold in solution than individual fouling, and a mixture of 100 ppm of PAX and 300 ppm of MIBC. It achieved the residual gold of 13.4 ppm followed by 300 ppm of PAX (12.84 ppm), a mixture of 100 ppm of PAX and 300 ppm of MIBC (11.62 ppm), 300 ppm of MIBC (7.48 ppm), 100 ppm of PAX (6.86 ppm), and lastly 100 ppm of MIBC (4.98 ppm). This is for 10 g of activated carbon.

The increase in carbon concentration tends to lower the fouling effect caused by a combination of PAX and MIBC. The increase in carbon concentration from 1 g to 10 g led to a decrease in residual gold from 13.4 ppm to 5.1 ppm for 100 ppm of MIBC and 300 ppm of PAX, and 11.62 ppm to 3.19 ppm for 100 ppm of PAX and 300 ppm of MIBC. This was almost the same as individual fouling of PAX and MIBC. The increase in carbon concentration from 1 g to 10 g led to a decrease in residual gold from 12.84 ppm to 4.73 ppm for 300 ppm of PAX and 7.48 ppm to 3.74 ppm for 300 ppm of MIBC. Likewise, the increase in carbon concentration from 1 g to 10 g led to a decrease in residual gold from 6.86 ppm to 2.84 ppm for 100 ppm of PAX and 4.98 ppm to 1.07 ppm for 100 ppm of MIBC. Although the increase in carbon concentration lowers the residual gold in the system, the mixture of 100 ppm of MIBC and 300 ppm of PAX leads to a higher residual gold than the individual fouling of either PAX or MIBC.

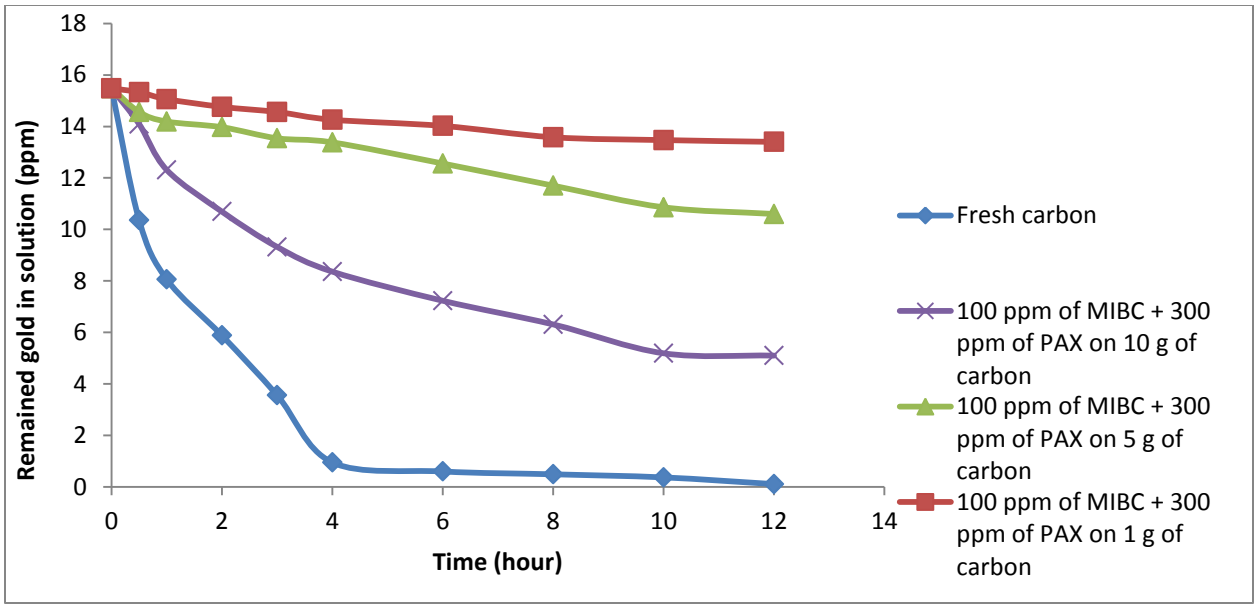


Figure 4.16: Residual gold during the carbon activity test for fresh carbon and carbon fouled by 100 ppm of MIBC and 300 ppm of PAX

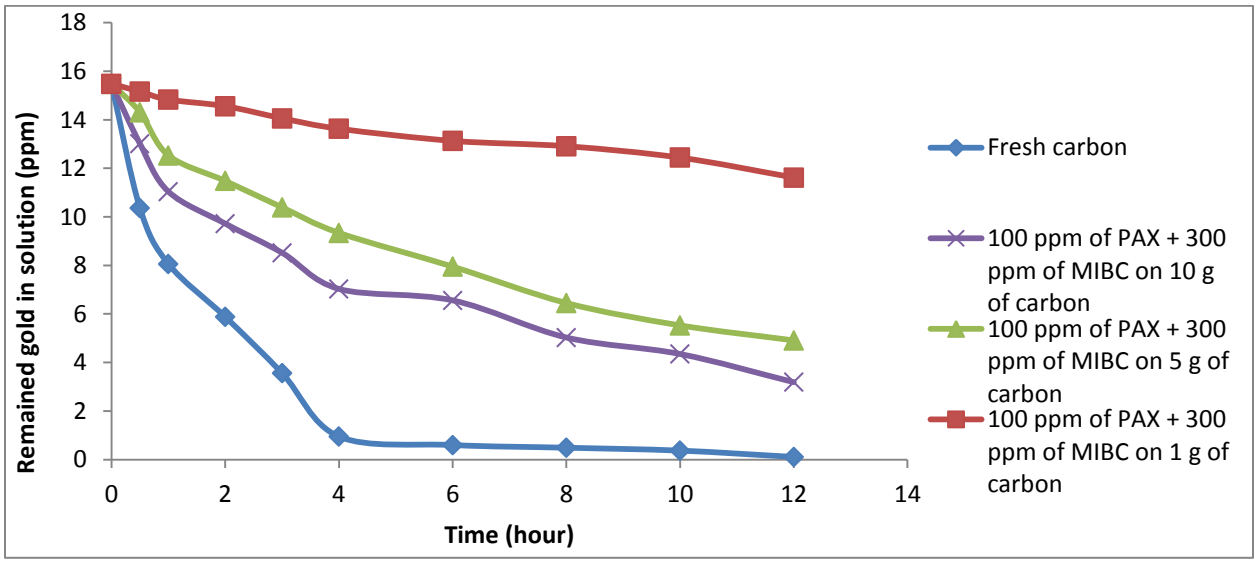


Figure 4.17: Residual gold during the carbon activity test for fresh carbon and carbon fouled by 100 ppm of PAX and 300 ppm of MIBC

Equation 2 was used to determine the loading capacity of gold onto activated carbon. Figure 4.18 and 4.19 show the loading capacity of fresh carbon, and carbon fouled by 100 ppm of MIBC and 300 ppm of PAX, and 300 ppm of MIBC and 100 ppm of PAX respectively. From the figures, it

can be observed that non-fouled activated carbon achieved a loading capacity of 2379 ppm whereas carbon fouled by a combination of 100 ppm of MIBC and 300 ppm of PAX achieved the loading capacity of 322 ppm, 755 ppm and 1606 ppm for 1 g, 5 g and 10 g of activated carbon respectively. Likewise, the use of 300 ppm of MIBC and 100 ppm of PAX results in a decrease in gold loading to 598 ppm, 1636 ppm and 1902 ppm for 1 g, 5 g and 10 g of activated carbon respectively.

A mixture of 300 ppm PAX and 100 ppm MIBC showed a lower loading capacity compared to the individually fouled activated carbon. The mixture showed a loading capacity of 1606 ppm for 10 g of activated carbon whist the individual fouling of 300 ppm of PAX and 300 ppm of MIBC showed a loading capacity of 1664 ppm and 1817 ppm respectively. However, the increase in carbon concentration tends to an increase the loading capacity of the fouled activated carbon. An increase in carbon concentration from 1 g to 10 g led to an increase in the loading capacity from 322 ppm to 1606 ppm for 100 ppm of MIBC and 300 ppm of PAX, and 598 ppm to 1902 ppm for 300 ppm of MIBC and 100 ppm of PAX. This was the same effect as in the individual fouling of PAX and MIBC where an increase in carbon concentration results in an increase in the loading of gold onto activated carbon.

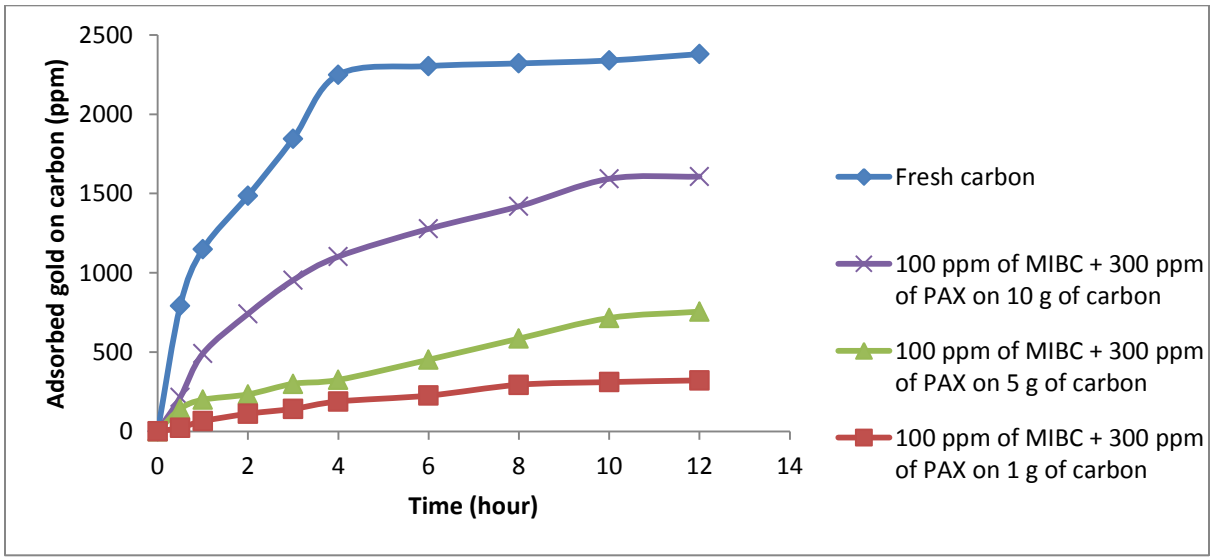


Figure 4.18: Loading capacity of fresh carbon and carbon fouled by 100 ppm of MIBC and 300 ppm of PAX

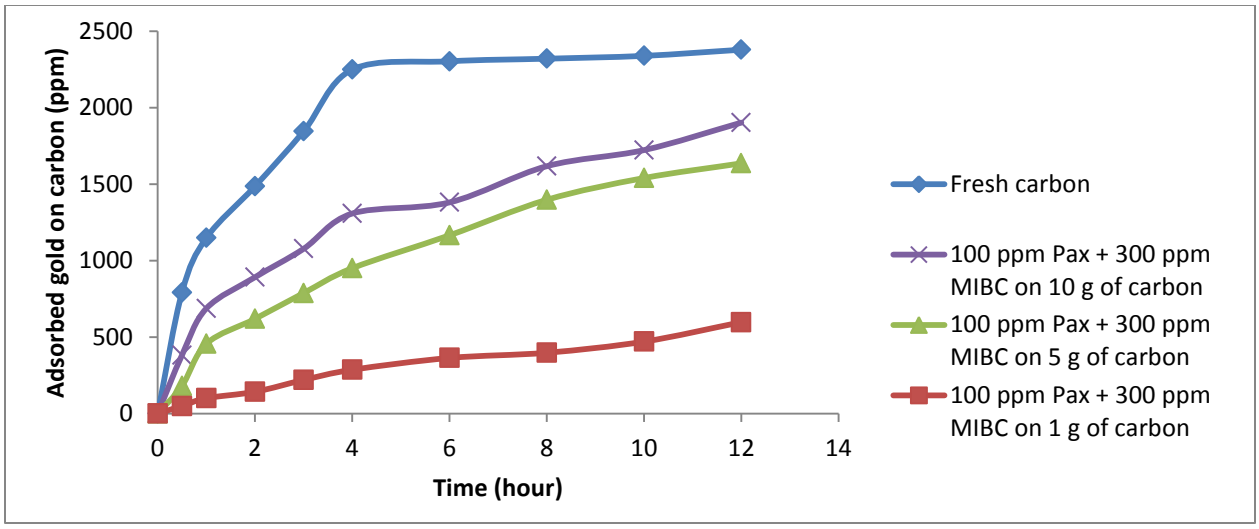


Figure 4.19: Loading capacity of fresh carbon and carbon fouled by 300 ppm of MIBC and 100 ppm of PAX

4.3.6 Effect of a Mixture of PAX and MIBC on the Adsorption Kinetics

The empirical kinetic equation (from equation 3), was used to determine the kinetic activity of activated carbon fouled by a combination of PAX and MIBC. Figure 4.20 and 4.21 illustrate the k-values for fresh carbon and carbon fouled by 100 ppm of MIBC and 300 ppm of PAX, and 300 ppm of MIBC and 100 ppm of PAX respectively.

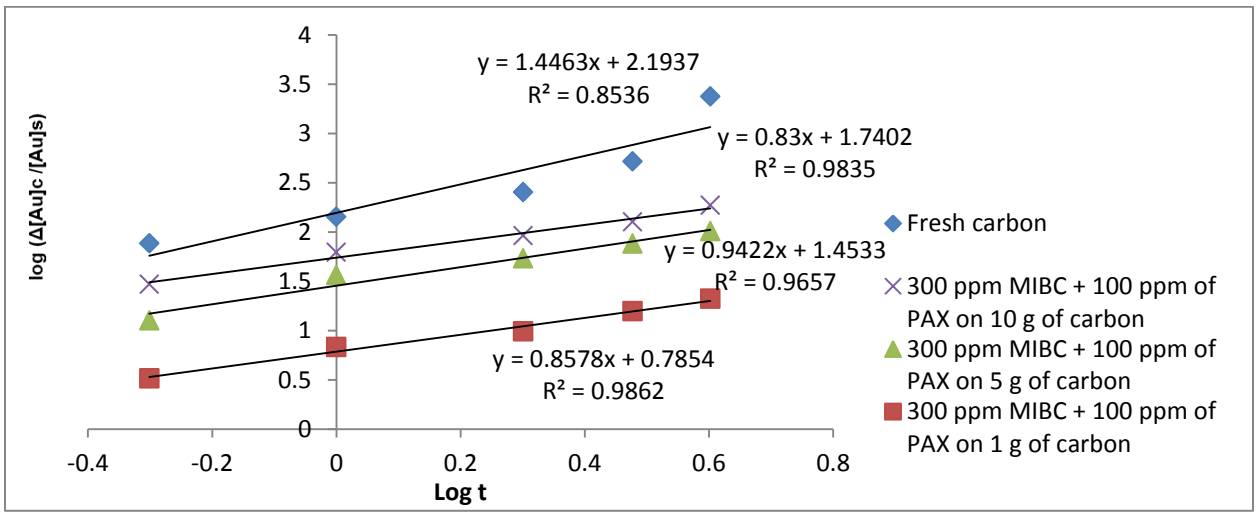


Figure 4.20: Plot of log ($\Delta[Au]_c^t / [Au]_s^t$) vs log t for fresh carbon and carbon fouled by 300 ppm of MIBC and 100 ppm of PAX

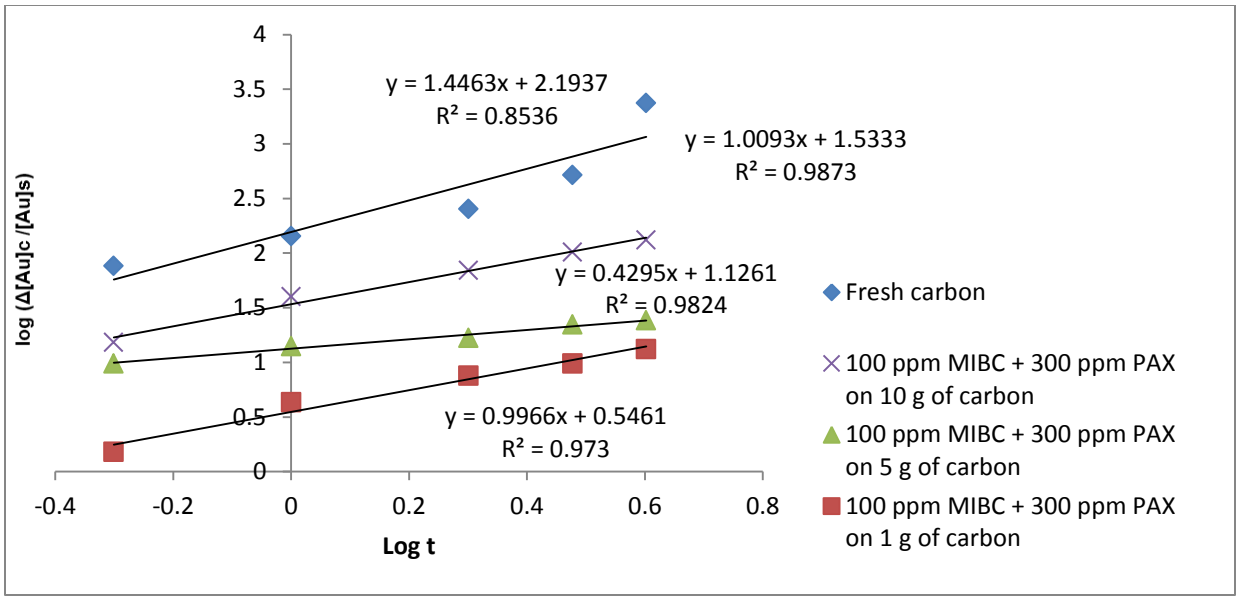


Figure 4.21: Plot of log ($\Delta[Au]_c^t$ /$[Au]_s^t$) vs log t for fresh carbon and carbon fouled by 100 ppm of MIBC and 300 ppm of PAX

A summary of the k values calculated from the information given in Figure 4.20 and 4.21 is shown in Table 4.6.

Table 4.6: k-values for the loading rate of gold onto non-fouled carbon and a mixture of PAX and MIBC fouled activated carbon

	Non-fouled activated carbon 1g	Fouled carbon by 100 ppm of MIBC and 300 ppm of PAX			Fouled carbon by 300 ppm of MIBC and 100 ppm of PAX		
		1g	5g	10g	1g	5g	10g
K –value (h^{-1})	156	4	13	34	6	28	55

Non-fouled activated carbon showed a higher k-value compared to the fouled activated carbon. Non-fouled activated carbon showed a k-value of 156 h^{-1}. Carbon fouled by a mixture of 100 ppm of MIBC and 300 ppm of PAX showed a k-value of 4 hr^{-1}, 13 hr^{-1} and 34 hr^{-1} for 1 g, 5 g

and 10 g of activated carbon respectively. Likewise, carbon fouled by a mixture of 300 ppm of MIBC and 100 ppm of PAX showed a k-value of 6 hr^{-1}, 28 hr^{-1} and 55 hr^{-1} for 1 g, 5 g and 10 g of activated carbon respectively. However, the increase in carbon concentration tends to increase the k-value. The increase in carbon concentration from 1 g to 10 g led to an increase in the k-value from 4 hr^{-1} to 34 hr^{-1} for 100 ppm of MIBC and 300 ppm of PAX, and 6 hr^{-1} to 55 hr^{-1} for 300 ppm of MIBC and 100 ppm of PAX. Although the increase in the carbon concentration increases the k-value for the gold loading onto activated carbon, the mixture of 100 ppm of MIBC and 300 ppm of PAX possess a lower k-value compared to individual fouling of PAX and MIBC.

4.4 Overall Discussion

The study revealed that the presence of flotation reagents (PAX and MIBC) in the gold adsorption circuit has a negative effect on the adsorption of gold onto activated carbon. These flotation reagents have an effect on the adsorption rate and loading capacity. Although the fouling effect of PAX and MIBC can be explained by same mechanism due to their hydrophobic nature, the fouling effect caused by a larger molecular weight compound is more serious than that caused by a smaller molecular weight compound (Fisher, 2000; Mahapatra, 2009). Table 4.7 summarizes the residual gold, loading capacity of gold and the k-value of gold loading onto non-fouled and fouled activated carbon at different concentration of flotation reagents.

Table 4.7: The residual gold, loading capacity of carbon for gold and the k-value for gold loading onto non-fouled and fouled activated carbon at different concentration of flotation reagents.

Flotation reagents concentration	Activated carbon dosage (g)	Residual gold (ppm)	Gold loading capacity (ppm)	k-value (h^{-1})
Non-fouled activated carbon (No flotation reagents)	1	0.11	2379	156
100 ppm PAX	1	6.86	1335	9
	5	4.16	1752	25
	10	2.84	1957	39
300 ppm PAX	1	12.84	408	9
	5	7.94	1167	12
	10	4.73	1664	22
100 ppm MIBC	1	4.98	1625	28
	5	3.85	1800	61
	10	1.07	2231	73
300 ppm MIBC	1	7.48	1238	20
	5	5.12	1604	46
	10	3.74	1817	56
300 ppm PAX and 100 ppm MIBC	1	13.4	322	4
	5	10.6	755	13
	10	5.1	1607	34
300 ppm MIBC and 100 ppm PAX	1	11.62	598	6
	5	4.91	1636	28
	10	3.19	1902	55

4.4.1 Effect of Flotation Reagents on the Residual Gold

From Table 4.7, it can be observed that fouled carbon has a high residual gold remaining in solution than non-fouled carbon. Petersen *et al*. (1997) showed that, high residual gold may be due to the adsorption of flotation reagents on the surface of activated carbon and not into the pores of activated carbon. The adsorption decreases the number of active sites on the carbon surface and results in high residual gold. The adsorption is due to difficult in the diffusion of bigger molecules of flotation reagents into the pores (Salarirad *et al*., 2011). However, the average size of flotation reagents such as PAX and SEX are 4.9 and 5.4 nm respectively (Petersen *et al*., 1997). Their sizes are very small compared to diameters of micropores and mesopores. A micropore has a diameter of less than 10 nm whereas mesopores has a diameter of 100 nm (TIGG Library, 2015). This indicates that flotation reagents may be adsorbed into the pores of activated carbon. Likewise, work done by Petersen *et al*. (1997) and Salarirad *et al*. (2011) did not study the structure of the activated carbon; surface and internal structure. The study of activated carbon structure enables the observation of the presence of particles inside the pores of activated carbon, and changes on the internal structure of carbon (surface area, pore size and pore volume) (Mahapatra, 2009). In this study, the adsorption of flotation reagents inside the pores of activated carbon was observed through SEM and BET analysis. Therefore, the study does not agree with the work by Petersen *et al*. (1997) and Salarirad *et al*. (2011).

Young *et al*. (1991) and Ross (1993) in contrast to the work by Petersen *et al* (1997) however, suggest that the high residual gold may be due to the adsorption of flotation reagents molecules on the surface and pores of activated carbon. This work agrees with the studies by Young *et al*. (1991) and Ross (1993).

High residual gold may also be explained by fewer active sites on fouled activated carbon as a result of adsorption of elements of PAX and MIBC on the surface and pores of activated carbon. The adsorption of elements occurs due to lack of selectivity of activated carbon (Mahapatra, 2009). It reduces the quantity of active sites on the surface and pores of carbon available for the adsorption of gold. Thus, fouled carbon will possess fewer active sites than non-fouled carbon. To support this assumption, EDS analysis (Table 4.2 and 4.3) show the presence of elements on the surface structure and pores of fouled activated carbon. In addition, BET analysis (Table 4.1)

shows a decrease in the surface area and pore volume of fouled carbon as a result of adsorption of elements of PAX and MIBC. This indicates that fouled activated carbon have the ability to accommodate a smaller amount of gold than non-fouled carbon due to smaller surface area and smaller volume.

4.4.2 Effect of Flotation Reagents on the Carbon Loading Capacity for Gold

From Table 4.7, it can be seen that fouled carbon has a lower loading capacity than non-fouled carbon. Salarirad *et al*. (2011) showed the ability of 4.5 g of activated carbon to load up to 5000 ppm of the dissolved gold. In this study, the poor loading capacity of fouled activated carbon may be explained by the adsorption of flotation reagents on the surface and pores of activated carbon (Young *et al*., 1991; Ross, 1993).

Virgin activated carbon possesses a large number of adsorption sites which are located at the edge defects in the graphite structure of activated carbon (Mahapatra, 2009). The adsorption of gold and flotation reagents both take place on the adsorption sites of activated carbon. This indicates that the adsorption of flotation reagents results in a decrease in the number of adsorption sites on the surface and in the pores of activated carbon which consequently leads to poor loading of gold (Mahapatra, 2009). To support this assumption of adsorption of flotation reagents, SEM images (Figure 4.1, 4.2 and 4.3) and EDS analysis (Table 4.2 and 4.3) show the presence of particles/elements inside the pores of fouled activated carbon. Likewise, BET analysis (Table 4.1) shows a decrease in the pore volume and surface area of fouled carbon. This suggests that non-fouled carbon possesses a larger number of adsorption sites compared to fouled carbon as a result of zero adsorption of flotation reagents.

Salarirad *et al*. (2010) argued that the same loading capacity can be achieved by both fouled and non-fouled activated carbon. This was due to an increase in the residence time whereby both fouled and non-fouled carbon achieved same loading capacity of 3870 ppm after 6 hrs and 15 hrs respectively. This suggests that a higher loading capacity to the levels acceptable at a gold plant could have been achieved if a longer residence time had been used.

4.4.3 Effect of flotation reagents on the k-value for gold loading

From Table 4.7, it can be noticed that non-fouled carbon shows a higher k-value than fouled carbon. The presence of flotation reagents show a decrease in the k-value for gold loading onto activated carbon. Ibragimova *et al*. (2007) reported that in industrial gold sorption processes, the k-value should be kept above 80 - 100 hr^{-1}. However, Fisher (2000) and La Brooy *et al*. (1984) indicated that the acceptable k-value in gold industries is above 50 hr^{-1}. Operating below the acceptable limit will lead to gold losses as a result of poor mass transfer of gold onto activated carbon. In this study the k-value for gold loading onto non-fouled carbon, 5 g and 10 g of 100 ppm MIBC fouled carbon, 10 g of a mixture of 300 ppm of MIBC and 100 ppm of PAX, and 10 g of 300 ppm MIBC fouled carbon were the only ones found to be above the acceptable level. The rest of the fouled carbon was shown to be below the acceptable level.

The loading of flotation reagents colonizes a larger area inside the pores and results in the reduction of pore sizes of fouled carbon (Mahapatra, 2009). The reduction in pore size results in a difficult in the diffusion of gold molecules into the pores of fouled carbon. The results show that a lower loading of flotation reagents is associated with a higher mass transfer than a higher loading of flotation reagents. This indicates that the rate of gold diffusion into the pores of fouled activated carbon will be much lower than for non-fouled carbon. To support this assumption, SEM images (Figure 4.1, 4.2 and 4.3) show the presence of particles inside pores of fouled activated carbon. BET analysis (Table 4.1) also shows a decrease in the pore size of fouled carbon as a result of loading/deposition of larger molecules of flotation reagents inside the pores of activated carbon

Salarirad *et al*. (2011) reported that the poor kinetic activity is probably due to film formation on the external surface of activated carbon. This film acts as a barrier by reducing mass transfer of gold onto the pores of activated carbon (Salarirad *et al*., 2011). Flotation reagents such as potassium amyl xanthate and methyl isobutyl carbinol, do not penetrate into the pores of activated carbon and only form a layer on the superficial surface of the carbon granules that diminish mass transfer, resulting in reduced sorption kinetics and hence affect the adsorption rate of gold onto activated carbon (Salarirad *et al*., 2010). However, in this study the formation of layers on the external surface of activated carbon was not observed, and only the loading of

flotation reagents on the surface and pores of activated carbon was observed. This indicates that the results presented in this study do not completely agree with the work presented by Salarirad *et al*. (2010; 2011).

Mahapatra (2009) proposed that the poor k-value for gold loading may be due to the difficult of gold molecules penetrating into the pores as a result of loading of flotation reagents into the pores of fouled carbon. The work by Mahapatra (2009) and Ross (1993) agrees with the results in the study in which the loading of flotation reagents into the pores of fouled activated carbon was observed through SEM and EDS analysis.

The results in this section show that although flotation is a necessary pre-cyanidation step required to recover gold which is finely disseminated in sulphide minerals, it is necessary to keep the k-value above the acceptable limit. This can be achieved by using an optimum dosage of flotation reagents in the circuit. Likewise, it can be achieved by the removal of flotation reagents from the pulp before entering into the adsorption circuit. Bilston *et al*. (1990) recommended the use of pre-treatment methods such as oxidation, filtration, and the combination of both methods. The methods aim to displace or remove flotation reagents present on the mineral surface of gold concentrate. This tends to lower the amount of flotation reagents reporting to the adsorption circuit. Moreover, BHP engineers (1989) showed the advantage of using roasting on the removal of flotation reagents present on the mineral surface of gold concentrate. These results therefore, suggest that, optimum dosage of flotation reagents followed by the removal of flotation reagents present on the mineral surface before gold cyanidation, to be the best solution to keep the k-value above the acceptable limit.

4.4.4: Effect of a mixture of PAX and MIBC

Activated carbon fouled by a mixture of PAX and MIBC shows high residual gold, poor loading capacity and poor k-value of gold loading onto activated carbon. The adsorption of the reagents lowers the number of active sites of activated carbon and results in gold losses in the circuit (Mahapatra, 2009; Ross, 1993). The effect was more critical for an increase in PAX concentration than for MIBC concentration. This can be explained by the effect of a larger molecular weight compound (PAX) which takes possession of large number of active sites of activated carbon than lower molecular weight compounds (Madan and Prakash, 1987;

Mahapatra, 2009; Davidson, 1992). This shows that the mixture of PAX and MIBC occupies many active sites of activated carbon and results in fewer sites available for gold adsorption.

4.4.5: Comparison between PAX and MIBC Fouled Carbon

A comparison between the effect of PAX and MIBC on deactivation of activated carbon was undertaken. From Table 4.7, it can be noticed that the effect of flotation reagents on carbon activity was found to be more significant with increasing PAX concentration than with increasing MIBC concentration. The effect resulting from an increase in PAX concentration is probably due to a larger molecular weight of PAX. PAX has a molecular weight of 164.289 g/mol whereas MIBC has a molecular weight of 102.2 g/mol (Petersen *et al*., 1997). The effect of molecular weight was studied by Traube (1891) and led to the introduction of Traube's rule (Fisher, 2000). This rule predicts that, the longer the chain molecule in the homologous series, the stronger is the adsorption onto activated carbon. Likewise, Young *et al*. (1991) showed that the loading of xanthate onto carbon was higher than gold loading. This suggests that the adsorption of a larger molecular weight compound (longer chain molecules) is much stronger than that of smaller molecular weight compounds (Rees, 2000; Mahapatra, 2009). The stronger adsorption is due to the fact that larger molecular weight compounds have stronger Van der Waals forces of attraction (Madan and Prakash, 1987; Davidson, 1992). Larger number of electrons in a molecule results in a greater diffusion of electron clouds. This results in a greater polarization and hence an increase in the Van der Waals forces. Since molecular weight is assumed to be direct proportional to the number of electrons in a molecule, the Van der Waals forces increase with an increase in molecular weight (Madan and Prakash, 1987). To support this assumption, EDS analysis (Table 4.2 and 4.3) showed a high composition of elements in PAX fouled carbon than MIBC fouled carbon. In addition, SEM images (Figure 4.1, 4.2 and 4.3) showed the presence of more particles inside the pores of PAX fouled carbon than MIBC fouled carbon. Additionally, BET analysis (Table 4.1) showed a significant decrease in the average area, pore volume and pore size of PAX fouled carbon than MIBC fouled carbon. This indicates that the available area for adsorption of gold onto PAX fouled carbon is much smaller than MIBC fouled carbon suggesting that the effect of PAX is much greater when mixed with MIBC. Likewise, it indicates that the rate of gold diffusion is smaller on PAX fouled carbon than MIBC

fouled carbon due to a significant decrease in pore size. The diffusion of gold molecules is much faster on pores which contain few molecules than pores which contain more molecules. Although the use of flotation reagents with higher molecular weight results in a significant fouling of activated carbon, it is however, mostly used in gold plants due to its high recovery compared to the use of lower molecular weight reagents (Wills, 2006; Fisher, 2000).

4.4.6: The Effect of Carbon Concentration

From Table 4.7, it can be seen that the increase in carbon concentration during the fouling stage seems to lower the fouling effect of flotation reagents. This is observed through the decrease in residual gold in solution, an increase in the loading capacity and an increase in the k-value for gold loading onto activated carbon. This may be explained by availability of more adsorption sites on the surface and pores of 10 g of fouled carbon than 1 g of fouled carbon. This is associated with the effect of the ratio factor of the amount of activated carbon to the concentration of flotation reagents. The concentration of flotation reagents was kept constant while varying the concentration of activated carbon. The ratio factor increases as a result of an increase in the concentration of activated carbon to 10 g and decreases as a result of a decrease in the concentration of activated carbon to 1 g. A decrease in the ratio factor results in a high loading of flotation reagents onto activated carbon. This reduces the number of adsorption sites available on the activated carbon leading to a high residual gold in solution, poor loading capacity and poor adsorption rate of gold. This indicates that more of the adsorption sites of 1 g of activated carbon will be occupied by flotation reagents than for 10 g of activated carbon. Mahapatra (2009) also reported that the use of high concentration of fouled activated carbon may results in low residual gold, high loading of gold and high adsorption rate. Although an increase in carbon concentration results in an increase in the operating costs, it is a possible solution for minimising gold losses in the circuit which subsequently leads to an increase in company revenue.

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATIONS

5.1. Introduction

Flotation reagents have been found to have a negative effect on the activated carbon used in the gold recovery process. The review of literature suggested that, employment of a low concentration of flotation reagents and a high concentration of the activated carbon decreases the fouling effect of flotation reagents. Therefore, the main objective of this study was to investigate the fouling effect of commonly used flotation reagents (PAX and MIBC) on the adsorption of gold by activated carbon.

The objectives of the study were:

- To investigate the effect of different levels of concentration of flotation reagents on the loading capacity of gold onto activated carbon.
- To investigate the effect of different levels of concentration of flotation reagents on the rate of gold adsorption onto activated carbon.

5.2 Effect of Flotation Reagents on the Loading Capacity of Gold onto Activated Carbon

Activated carbon is used to adsorb aqueous gold that is present in the leach circuit. The loading capacity of gold onto activated carbon depends on the type and concentration of flotation reagents present in the circuit. The results presented in this study have shown that the fouling effect increases with an increase in the concentration of flotation reagents. In addition, the use of larger molecular weight reagents (such as PAX) seems to cause more fouling of the activated carbon resulting in high losses of gold in the circuit. The results show that the use of 300 ppm of PAX results in a minimum loading capacity of 409 ppm for 1 g of carbon. Likewise, a mixture of 300 ppm of PAX and 100 ppm of MIBC resulted in a minimum loading capacity of 322 ppm.

The poor loading capacity was explained as due to adsorption of flotation reagents on the surface and pores of activated carbon. The adsorption of flotation reagents result in a decrease in the number of adsorption sites available for gold adsorption.

5.3 Effect of Flotation Reagents on the Rate of Gold Adsorption onto Activated Carbon

In order to evaluate the adsorption rate of activated carbon, the empirical kinetic equation was used to determine the k-value of gold loading onto activated carbon. The results indicated that flotation reagents have a significant effect on the k-value. The effect is more pronounced on the use of a mixture of 100 ppm of MIBC and 300 ppm of PAX. It decreases the k-value to 4 hr^{-1}. The flotation reagents were seen to decrease the k-value of gold loading below the acceptable level of 50 hr^{-1} except for 10 g of 300 ppm of MIBC fouled carbon, 5 g and 10 g of 100 ppm MIBC fouled carbon, and 10 g of a mixture of 300 ppm of MIBC and 100 ppm of PAX.

The decrease in the k-value was explained as due to the difficult associated with gold molecules in penetrating deep into the pores of fouled carbon. This occurs due to loading of PAX and MIBC molecules inside the pores, and hence, causing a decrease in the sizes of the pores.

5.4 Minimising the Fouling Effect of Flotation Reagents

The overall results showed that the fouling effect of flotation reagents can be minimized by the use of a low concentration of flotation reagents in the circuit. In addition, it can be minimized by a use of high concentration of activated carbon in the circuit. Although the increase in carbon concentration has an impact on the operational costs of the circuit, it increases the loading capacity of gold, and the k-value for gold loading onto activated carbon.

5.5 Recommendations

Based on the knowledge acquired in this study, the following recommendations are proposed for future studies:

- Investigation into the effect of an increase in residence time during the fouling stage and activity stage.
- Investigating the competitive adsorption between flotation reagents and gold onto activated carbon.
- The activity of non-fouled activated carbon should be compared to the activity of regenerated fouled carbon. The process will enable to check the capability of thermal regeneration on the removal of foulants.
- The fouling effect of flotation reagents should be investigated for a continuous process. The process will enable the assessment of the cumulative effect of flotation reagents on gold adsorption onto activated carbon.

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APPENDICES

APPENDIX A

BET Surface Area Report for Non-fouled Carbon and Fouled Carbon

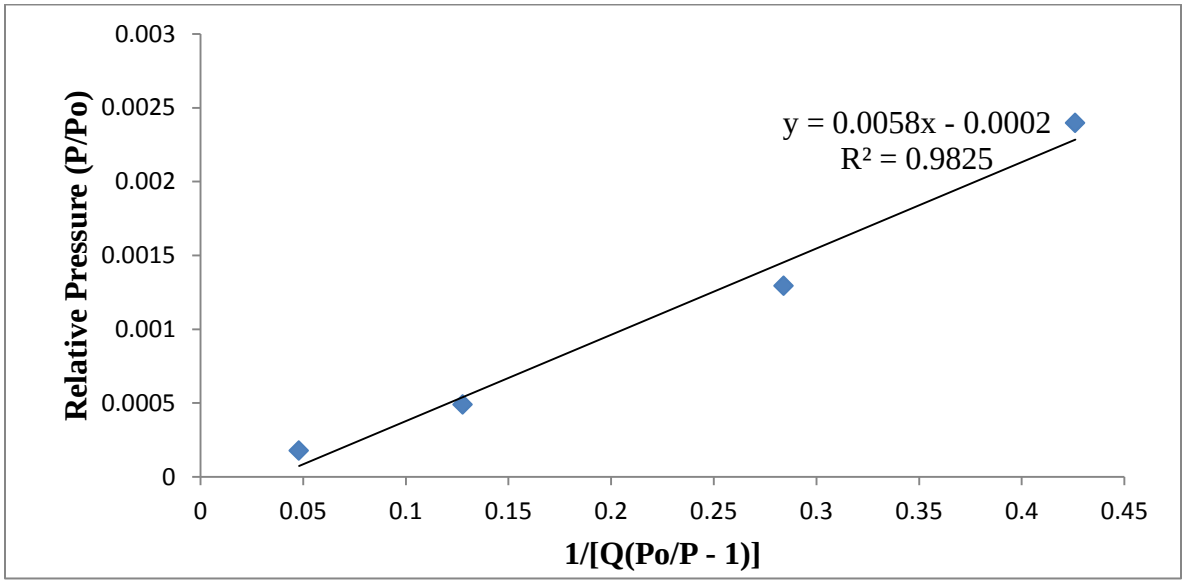

Figure A 1: BET surface area plot for non-fouled carbons

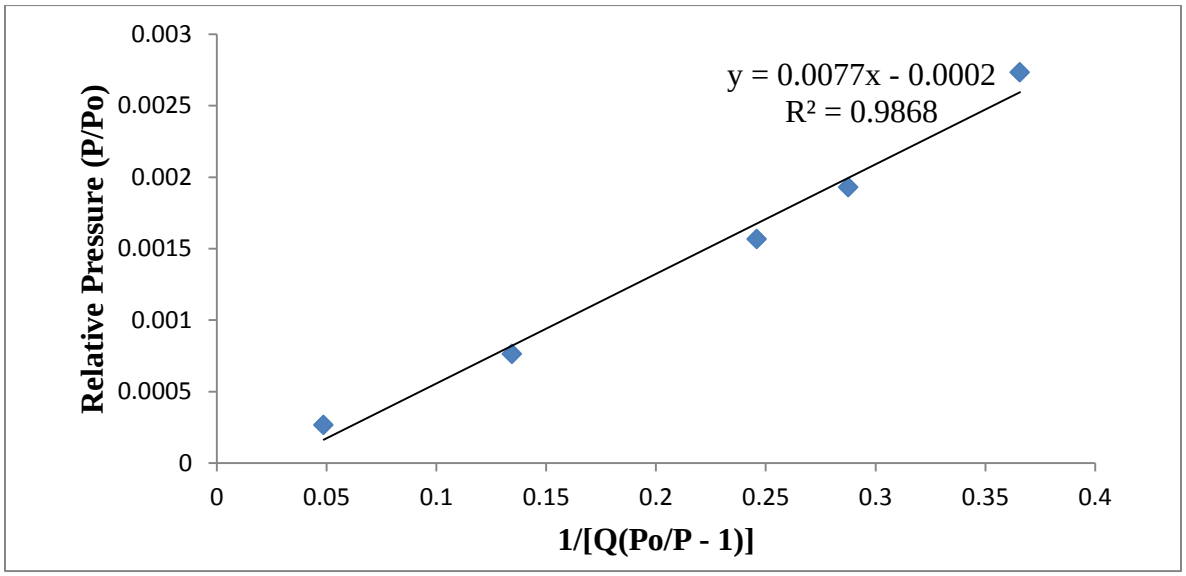

Figure A 2: BET surface area plot for MIBC fouled carbons

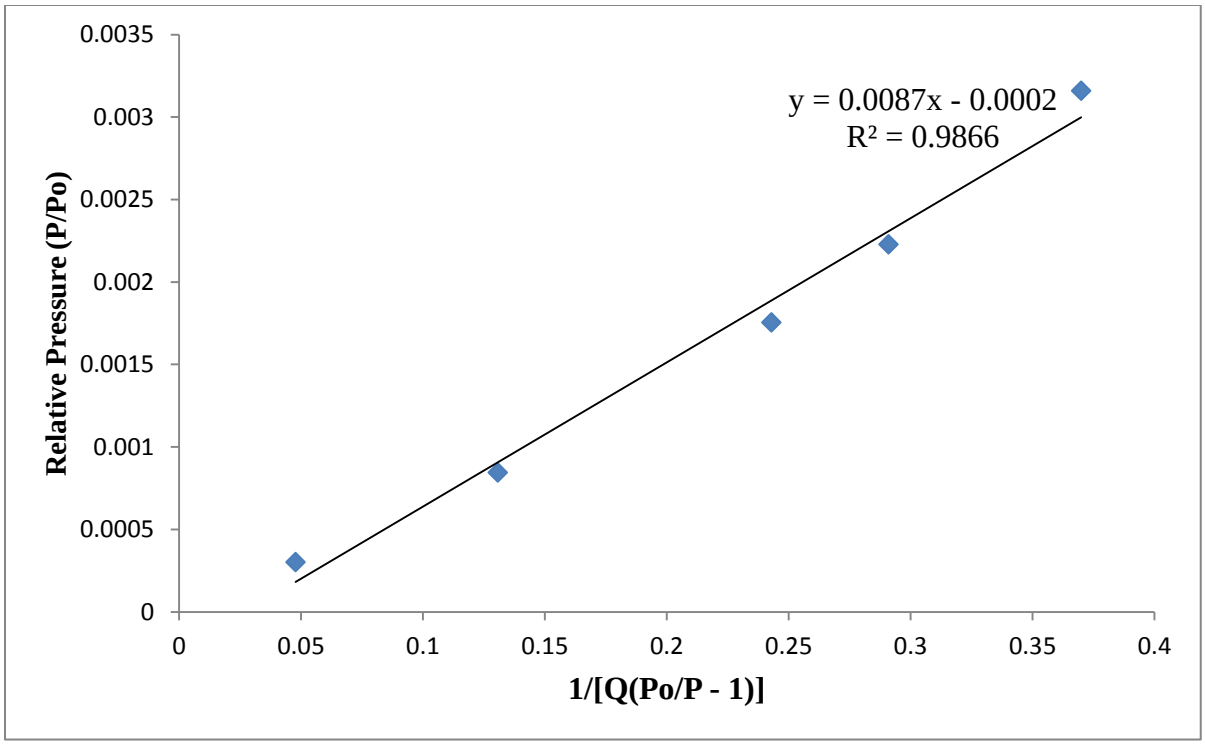

Figure A 3: BET surface area plot for PAX fouled carbons

APPENDIX B

Residual Gold Data

Table B 1: Residual gold data on non-fouled carbon and PAX fouled carbon

Time (hour)	Residual gold for Non-fouled carbon (ppm)	Residual gold for carbon fouled by 100 ppm of PAX (ppm)			Residual gold for carbon fouled by 300 ppm of PAX (ppm)		
	1g	1g	5g	10g	1g	5g	10g
0	15.48	15.48	15.48	15.48	15.48	15.48	15.48
0.5	10.36	15.06	14.83	14.55	14.98	14.89	14.53
1	8.06	14.61	12.53	10.84	14.66	14.37	13.73
2	5.88	13.89	10.22	8.83	14.38	13.12	11.71
3	3.56	12.85	9.18	7.21	13.98	12.67	10.16
4	0.95	11.25	7.05	6.07	13.85	12.03	8.75
6	0.6	10.08	5.92	4.95	13.73	11.23	6.78
8	0.49	8.94	5.94	4.05	13.39	9.84	5.95
10	0.37	7.84	4.8	3.43	13	8.37	5.15
12	0.11	6.86	4.16	2.84	12.84	7.94	4.73

Table B 2: Residual gold data on non-fouled carbon and MIBC fouled carbon

Time (hour)	Residual gold for Non-fouled carbon (ppm)	Residual gold for carbon fouled by 100 ppm of MIBC (ppm)			Residual gold for carbon fouled by 300 ppm of MIBC (ppm)		
	1g	1g	5g	10g	1g	5g	10
0	15.48	15.48	15.48	15.48	15.48	15.48	15.48
0.5	10.36	14.05	12.52	12.42	14.55	13.74	13.55
1	8.06	13.25	10.98	10.59	13.38	11.13	10.57
2	5.88	11.85	9.55	8.4	12.58	9.43	8.32
3	3.56	10.67	8.33	6.25	11.27	8.83	7.15
4	0.95	8.97	7.2	5.5	10.69	7.81	6.85
6	0.6	6.97	6.55	3.06	9.36	6.28	5.5
8	0.49	5.86	4.94	2.35	8.51	5.96	4.66
10	0.37	5.06	4.18	1.43	8.08	5.33	4.14
12	0.11	4.98	3.85	1.07	7.48	5.12	3.74

Table B 3: Residual gold data on non-fouled carbon and carbons fouled by a mixture of PAX and MIBC

Time (hour)	Residual gold for Non-fouled carbon (ppm)	Residual gold for carbon fouled by 100 ppm of MIBC and 300 ppm of PAX (ppm)			Residual gold for carbon fouled by 300 ppm of MIBC and 100 ppm of PAX (ppm)		
	1g	1g	5g	10g	1g	5g	10
0	15.48	15.48	15.48	15.48	15.48	15.48	15.48
0.5	10.36	15.33	14.56	14.09	15.16	14.31	13.01
1	8.06	15.06	14.19	12.31	14.83	12.53	11.04
2	5.88	14.76	13.97	10.69	14.56	11.48	9.72
3	3.56	14.56	13.54	9.32	14.06	10.39	8.52
4	0.95	14.26	13.38	8.36	13.63	9.34	7.03
6	0.6	14.02	12.56	7.23	13.13	7.95	6.56
8	0.49	13.58	11.7	6.31	12.91	6.45	5.03
10	0.37	13.47	10.86	5.19	12.44	5.53	4.35
12	0.11	13.4	10.6	5.1	11.62	4.91	3.19

APPENDIX C

Adsorption Kinetics Data

Table C 1: Adsorption kinetics data on non-fouled carbon and PAX fouled carbon

Log t (hr)	Non-fouled carbon (log $(\Delta[Au]^t_c /[Au]^t_s)$ 1g	Carbon fouled by 100 ppm of PAX ($\log (\Delta[Au]^t_c /[Au]^t_s)$			Carbon fouled by 300 ppm of PAX ($\log (\Delta[Au]^t_c /[Au]^t_s)$		
		1g	5g	10g	1g	5g	10g
-0.30103	1.8836	0.6351	0.8315	0.9953	0.7132	0.7877	1.0052
0	2.1538	0.9646	1.5616	1.8212	0.9374	1.0776	1.2951
0.30103	2.4026	1.2484	1.9013	2.0666	1.0734	1.4447	1.6975
0.477121	2.7145	1.5008	2.0262	2.2493	1.2203	1.5357	1.9087
0.60206	3.3743	1.7649	2.2674	2.3801	1.2605	1.6473	2.0757

Table C 2: Adsorption kinetics data on non-fouled carbon and MIBC fouled carbon

Log t (hr)	Non-fouled carbon (log $(\Delta[Au]^t_c /[Au]^t_s)$	Carbon fouled by 100 ppm of MIBC (log $(\Delta[Au]^t_c /[Au]^t_s)$			Carbon fouled by 300 ppm of MIBC (log $(\Delta[Au]^t_c /[Au]^t_s)$		
	1g	1g	5g	10g	1g	5g	10g
-0.30103	1.8836	1.1974	1.5634	1.5813	0.9953	1.2923	1.343389
0	2.1538	1.4158	1.8023	1.8541	1.3855	1.7817	1.856777
0.30103	2.4026	1.6759	1.9828	2.1155	1.5524	1.9970	2.124561
0.477121	2.7145	1.8437	2.1234	2.3590	1.7621	2.0666	2.25611
0.60206	3.3743	2.0505	2.2504	2.4485	1.8411	2.1819	2.290091

Table C 3: Adsorption kinetics data on non-fouled carbon and carbons fouled by a mixture of MIBC and PAX

Log t (hr)	Non-fouled carbon (log $(\Delta[Au]^t_c /[Au]^t_s)$	Carbon fouled by a mixture of 100 ppm of MIBC and 300 ppm of PAX (log $(\Delta[Au]^t_c /[Au]^t_s)$			Carbon fouled by a mixture of 300 ppm of MIBC and 100 ppm of PAX (log $(\Delta[Au]^t_c /[Au]^t_s)$		
	1g	1g	5g	10g	1g	5g	10g
-0.30103	1.8836	0.1803	0.990397	1.1838	0.5142	1.1023	1.4681
0	2.1538	0.6351	1.148378	1.6005	0.8315	1.5616	1.7941
0.30103	2.4026	0.8780	1.223551	1.8411	0.9903	1.7318	1.9625
0.477121	2.7145	0.9903	1.345954	2.0099	1.1940	1.8798	2.1019
0.60206	3.3743	1.1220	1.385534	2.1200	1.3224	2.0075	2.2696